



## Official Notices.

### REPORTS OF THE PROGRESS OF APPLIED CHEMISTRY.

Owing to unforeseen delays in the receipt of the manuscript of certain sections of these Reports, it is regretted that the publication of the first volume is unavoidably postponed until March.

### CROSS AND BEVAN ESSAY PRIZE OF £20 AND LATHAM RESEARCH GRANT OF £300.

The Council have pleasure in announcing that a gift of £20 has been made by Messrs. Cross and Bevan as a prize for an essay on "The Inter-connection of Economic Botany and Chemical Industry."

A further sum of £300 has also been presented by their friend, Mr. T. P. Latham, of Weybridge, to be paid in three annual instalments to such one of the competing essayists as may be judged to be of conspicuous ability, as a grant towards his expenses, on the condition that he apply himself, during three years, to research of approved character on a subject cognate with that discussed in his essay.

The immediate object of the donors is to promote the study of economic botany with special reference to its bearing on chemical industry, giving the widest possible interpretation of the relationship.

They desire, under the auspices of the Society, to assist in forming public opinion in this direction and to discover and further the career of a student of promise who may wish to devote himself to work in such a field.

The Essay Prize will be open to all members of the Society who are British subjects. The Research Grant will be limited to competitors under 25 years of age at the time of sending in the essay.

Essays are to be submitted to the Council of the Society not later than the close of the year 1917. The awards will be made promptly. Neither the prize nor the first grant will be given unless the essayist or essayists shall be deemed to have real merit. The Latham Research Student will be required to submit a report at the close of each year, to satisfy the Council that he has made and is likely to make proper use of the grant.

## London Section.

Meeting held at Burlington House on Monday, January 15th, 1917.

MR. A. R. LING IN THE CHAIR.

### A NEW METHOD OF EXTRACTING THE VAPOROUS CONSTITUENTS FROM COAL GAS.

BY R. LESSING, PH.D.

The recovery of benzol from coal gas is a problem which has engaged the attention of the carbonising industry for the last 50 years. A good account of its early development, in which men like Cusiter, H. Caro, J. Hardman, G. E. Davis, and P. Carves appear as pioneers, is given in Lunge's "Coal Tar and Ammonia."

The process was first practised in the English "carbonising" works, then taken up by the by-product coke oven industry, and reintroduced into this country by Continental coke-oven builders. The gas industry became interested in the recovery of benzol from gas a few years ago as a

means of augmenting the supply of motor fuel, but the statutory requirements of illuminating power stood in the way of its adoption on an appreciable scale.

Whilst the way had been paved, by the fixing in a few individual cases of a calorific standard—the more rational one in view of the growing importance of coal gas as a heating agent—it took the stern demands made by the present War to sweep away old traditions and idiosyncrasies and to make the valuation of gas on a calorific basis more general, or, at any rate, indemnify gas undertakings against deficiencies in illuminating power where they are caused by the extraction of benzol and toluol for munitions purposes. Under these conditions, created by the War, benzol extraction was taken up by many gas undertakings and to an increased extent by coke-oven works which had not previously practised it.

The principle of the process is practically the same in all plants although they may differ in details of design and working. It consists of scrubbing or washing the gas with an absorbent oil and subsequently distilling the absorbed hydrocarbons from the "benzolised" oil and returning the "debenzolised oil," after cooling, to the scrubbers or washers. The scrubbers used are in the main of three types: tower scrubbers filled with more or less efficient packing material; mechanical washers of the horizontal or vertical type; and bubbling washers of the Livesey type. As to stills, a great many varieties of design are in use, many of which are capable of improvement by the application of a more thorough study of the physico-chemical laws involved. Oils from coal tar, blast furnace tar, shale, and petroleum are being used as absorbents, the most important ones being creosote oil, green oil, and gas oil. Undistilled tar is also used as originally proposed by Friedlaender and Quaglio (Eng. Pat. No. 4888, April 1, 1887) and in that case the distilling plant can be dispensed with as fresh tar is produced at the works and the benzolised tar is worked up by the tar distiller. As the boiling range of tar is a very wide one it may act at the same time as a scrubbing and carburetting agent, and to low absorbing efficiency, on account of high water or carbon contents, is frequently added the risk of the gas picking up additional naphthalene from the tar.

The adoption of a process which dispenses with a distilling plant in preference to the more efficient method of washing with oil in a continuous cycle, exemplifies a certain justifiable reluctance on the part of managers of small or medium-sized gas works to add the duties of chemical manufacturers to their manifold responsibilities. It was, therefore, considered desirable to devise a process of benzol recovery which should combine extreme simplicity in design and working of the plant with a fair degree of efficiency.

The use of a "dry" scrubber filled with a solid absorbent material which would strip benzol from the gas without the employment of running wash oil, and from which the volatile products could be removed by steam distillation *in situ*, appeared to offer a solution of the problem.

I had observed a number of years ago in the analysis of spent oxide that it was difficult to obtain constant figures when endeavouring to estimate its moisture by passing a current of dry coal gas over it. I explained these results, even at that time, by the absorption of hydrocarbons and carbon disulphide in the gas by the free sulphur and particularly by the tarry matter in the spent oxide. On searching the literature I find that a similar observation had been made

with American spent oxide by Goodno (*Gas World*, 1893, 18, 296). However, I did not consider spent oxide a suitable material either for the absorption or for the distillation phase of the process. This opinion has since been confirmed with regard to the absorption of carbon disulphide on the practical scale by Teune in experiments carried out at the Amsterdam Gas Works (*J. Gas Lighting*, 1915, 130, 334).

It appeared to be necessary to find a material of greater and more definite absorptive power, and one which should offer considerably less back pressure than spent oxide would do and not be subject to chemical side-reactions.

It was found that the illuminating power of coal gas could be greatly reduced by passing it through a tube charged with pitch crushed to the size of a pea. It was, however, observed that the sharp-edged pieces became rounded, and that the pitch started "squatting" and finally "running" as soon as its viscosity had decreased sufficiently by the absorption of its solvents from the gas. This showed that it would be impossible to pack a scrubber with it, as it would tend to consolidate

allowing it to remain quiescent, and indeed to behave as a solid, as far as contact with the gas and the production of back pressure is concerned. The absorbed compounds can be liberated from their solution in the oil, *in situ*, by steam distillation, and to this phase of the steaming process apply the same considerations regarding the exposure of a large surface and avoidance of back pressure, as to the scrubbing period.

On this basis the following process was devised:—The gas is passed through a closed vessel which contains porous material soaked in a suitable oil—for instance, green oil or gas oil. The inert material may consist of broken highly porous brick or, preferably, of moulded pieces of definite shape, volume, weight, and available surface. The vaporous compounds in the gas in which the oil is soluble, *i.e.*, the hydrocarbons and organic sulphur compounds, which when isolated, are liquid at ordinary temperature, are absorbed. When the oil has taken up the required amount of the substance to be extracted, which will depend on the quantity and surface of the absorbing agent, the velocity and volume of the

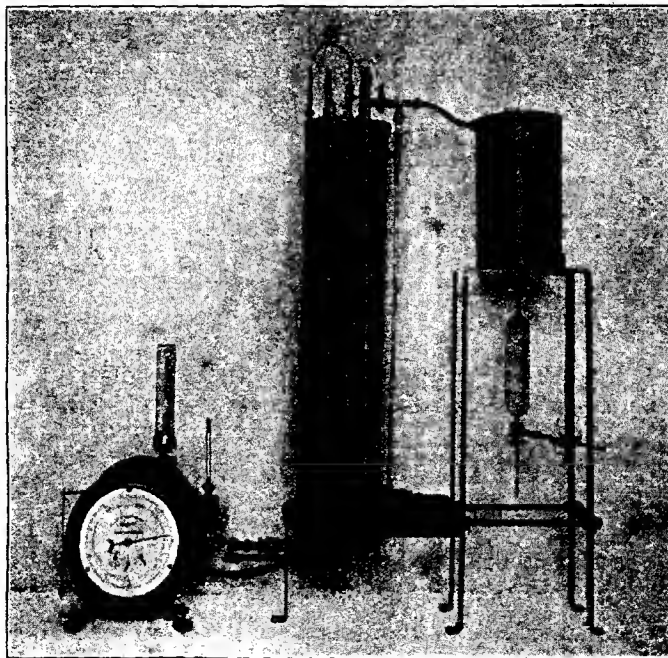


FIG. 1.

and block the passages, even if the temperature were kept low enough for the original pitch to remain rigid.

It was, therefore, necessary to supply a rigid support for the absorbent material. Whilst pitch had been selected on account of the high viscosity, both of itself and its solution in carbon disulphide, benzol, toluol, etc., it was found in the course of further experiments that oils of fairly low viscosity could be incorporated into highly porous inert materials without being unduly thinned or washed out by the solvent vapours from the gas. Whilst, therefore, in the usual methods of washing or scrubbing gas with oils the gas is caused to bubble through a body or pass through a shower or rain of washing liquid, it was found possible to charge scrubbers with oil carried on an inert and rigid support,

gas, the temperature and degree of its saturation, the mutual solubility of the vapours and the absorbing oil, and the vapour pressure of the solution formed, the gas inlet and outlet valves are closed, the gas current being directed into the second vessel of the series. Steam is then blown through the material, carrying the vapours of the absorbed compounds with it to the water-cooled condenser and finally to a receiver fitted with overflow pipes for water and crude benzol respectively. It is advisable to provide the "scrubber-still" with a jacket to avoid the condensation of steam, particularly on the walls. It is, however, not necessary to raise the whole of the inert carrying material to the temperature of distillation. As the oil forms a very thin film and is, therefore, completely exposed to the passing steam, the

volatile matter dissolved in it is distilled off before the non-conducting support is raised throughout its mass to the same temperature.

When the distillation is finished the steam is shut off and the vessel is cooled by passing water through the jacket, and is then ready for the next scrubbing period.

By employing three or more sets of "scrubber-stills" they can be worked in rotation, a definite schedule being arranged for the three periods of scrubbing, steaming, and cooling.

It will be seen that the labour involved is restricted to the manipulation of valves for changing over from gas to steam and *vice versa*, and to the collection of the distillate. As there are no pumps or other moving parts involved in the process, nor adjustments of the flow of oil, the plant does not require any attention except for the change-over from one period to another. The steaming is to be done only once in 24 hours, or at even longer intervals, and occupies only a fraction of the time available for scrubbing. The ground-space required is, if anything, smaller than that occupied by the usual combination of scrubbing and distilling plant. Losses of oil are restricted to the portion distilled off with the steam, particularly if superheated steam is used. In places where the crude benzol is fractionated this oil can be returned in the form of residue. Elsewhere it is replaced by fresh oil, which is occasionally introduced through the top of the scrubber and is allowed to soak into the material on its way to the bottom, where any excess is drawn off through drain-cocks.

The efficiency of scrubbing or washing in the customary manner depends upon constantly breaking up the oil into small drops or thin films, in order to expose new surfaces, and likewise upon deflecting the gas currents at short and frequent intervals to avoid the formation of inactive cores in bubbles or streams. In the proposed method all the oil is exposed in a thin film into the body of which the gas can readily diffuse and which, therefore, does not require breaking up or turning over.

Thickening of the wash oil in ordinary scrubbers is a disadvantage on account of the less intimate contact caused by a rise in viscosity. In this process, provided the solubility is not impaired, the thickening of the oil is rather an advantage, as a more viscous oil is more readily retained by the porous material than a thinner one; whilst the area of the oil film exposed to the gas and steam respectively remains unaltered.

The interaction of gas and oil further depends on time-contact. Assuming equal rates of gas passage and equal scrubbing surfaces of a "dry" scrubber and an "irrigated" scrubber a certain volume of gas would remain in contact with oil for about the same time in both cases. On the other hand the oil in the "dry" scrubber will remain in contact with gas throughout the whole scrubbing period, whilst the contact of each particle of oil passing through the "irrigated" scrubber or through a washer depends on the rate of flow, which varies in wide limits with different constructions, but it is always considerably shorter than that of the quiescent body of oil locked up in the "dry" scrubber. Consequently the oil in the latter has a chance to saturate itself with the vapours from the gas to the maximum limit of the permissible vapour pressure of the solution formed. The latter is more concentrated near the gas inlet than towards the outlet, and if the vessel is divided into compartments, or several vessels are arranged in series, one will be completely saturated whilst the following ones act as check vessels and it is only necessary to steam the completely saturated vessel whilst the second and third become Nos. 1 and 2 respectively, in the series.

Pending the elaboration and thorough testing of the process on a works scale, I have constructed an experimental apparatus for analytical and

research purposes based on the same principle, which has proved useful in this class of work during the last two years. The need for a simple and reliable method of controlling oil washing operations has been expressed on various occasions lately, and I have, therefore, been encouraged by the Ministry of Munitions to publish the details.

None of the purely chemical methods which have been proposed from time to time has been found satisfactory for the control of benzol extraction plants in gas and coke works. The most accurate physical method is probably the freezing out of the condensable hydrocarbons. This was proposed by St. Claire Deville (*J. des Usines & Gaz.* 1889), who extracted benzol and its homologues by passing from 10 to 20 cub. ft. of gas through glass coils placed in a freezing mixture at  $-22^{\circ}\text{C}$ . and weighing the condensate. He also determined the quantity of benzol required to saturate the gas at that temperature by further cooling it to  $-70^{\circ}\text{C}$ .

Lebeau and Damiens (*Comptes. Rendus*, 1913, 156, 144, 325) devised a method for the fractional distillation of liquefied coal gas on the lines of the researches of Ramsay and Travers. This method was amplified by Burrell, Seibert, and Robertson (*U.S. Bureau of Mines Tech. Paper* 104, 1915).

Whilst exceedingly useful for the estimation of the permanent gases and the total quantity of vapours—if that distinction be permitted—these methods deal with quantities too small to allow of a separation of the vapour mixture into its components. These are, therefore, lumped together into one figure representing "compounds having an inappreciable vapour-pressure at  $-78^{\circ}\text{C}$ ," and their volume in uncondensed form amounts to only about 1.5% of the total. Their further treatment is, therefore, out of the question.

H. F. Coward and F. Bailey (*Manch. Lit. and Phil. Soc.*, 1916, XXIV.) pass a current of gas through a tube immersed in a mixture of solid  $\text{CO}_2$  and ether for the purpose of estimating the illuminating power of the stripped gas on the lines of St. Claire Deville's experiments.

Whilst all these methods are very valuable for purely research purposes the apparatus is complicated and costly, and requires skilled manipulators; besides, the quantities of liquid hydrocarbons obtainable in reasonable time are too small for practical purposes.

The method most widely adopted for works purposes is the passage of gas through a train of wash bottles charged with tar oils or petroleum oils of various description. The gas is passed at the rate of about 1 cub. ft. per hour. The first bottle in the series is emptied twice a day, charged with fresh oil and placed at the end. The oil from all the bottles is collected and distilled in a metal retort, with the aid of steam for the fractions above  $130^{\circ}\text{C}$ . On account of the back-pressure caused by the oil and the low scrubbing efficiency of most types of wash bottles, the rate of gas cannot be increased, and a complete test of 100 cub. ft. of gas consequently takes practically 5 days.

On account of the bubbling the stream is not constant, and if the gas is to be tested for calorific value or illuminating power it has to be collected in a holder to obtain a steady flame. At best, absorption in wash bottles is attended by the drawbacks of rubber connections and leaks in stoppers.

The method described below overcomes these difficulties. Figure 1 is a photographic view, and Figure 2 is a diagrammatic section of the apparatus. The container, A, is filled with the oil-soaked inert material, C, which rests on a perforated bottom, B, and exerts only a negligible back-pressure to the gas. The gas to be tested enters through stop-cock, D, passes downwards through the material, and through the gas outlet, E, into the gas meter. I prefer to arrange the

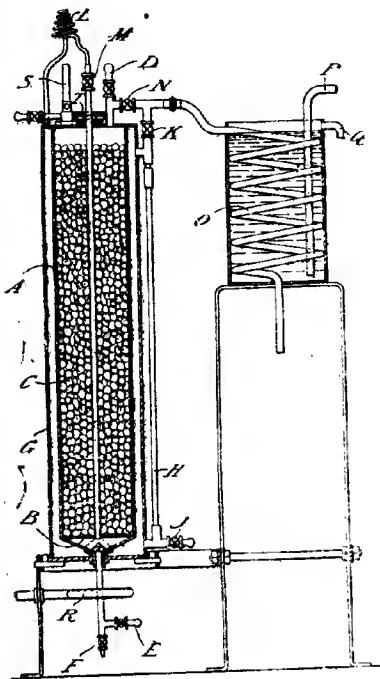


FIG. 2.

gas meter after the scrubber in order to avoid condensation or extraction of vapours in the meter. In most cases it is also more convenient to relate the results to totally extracted gas as a basis. The use of rubber tubing should be avoided as far as possible.

After leaving the meter the gas may be used for any further chemical or physical examination required. Or else it may be burned and then indicates, by its almost entirely blue flame, the satisfactory working of the scrubber. It burns with a perfectly steady flame, and if the burner is arranged on the lines of a jet photometer the length of flame, when compared with that of the unstripped gas, will give a direct, though somewhat empirical, measurement of the extracted hydrocarbons. Only the inner cone of the flame shows a small amount of luminosity, which is due to the non-condensable illuminants, such as ethylene, propylene, and acetylene, and to the small amount of vapour due to the vapour pressure of the solution formed, which is constant for any given temperature.

The container, A, is surrounded by a jacket, G, which is provided with a gauge, H, and drain-cock, J, and is filled with water during the scrubbing period. The gas is passed through the apparatus at the rate of about 5 cub. ft. per hour, and about 100 cub. ft. are used for one experiment. This will, therefore, cover a period of 20 hours, during which the apparatus does not require any attention at all.

Stop-cocks, E and D, are then closed, K is opened and the water run out through drain-cock, J, until it fills only about one-third of the jacket. A gas ring burner, H, is then lit and steam is raised in the jacket, the excess steam escaping through the condenser O. When the water is well boiling K is shut and stopcocks, M and N, are opened, the pressure having been relieved from time to time through N. At the same time the Bunsen burner,

S, is lit. The steam raised in the jacket passes now through the super-heater coil, L, and stop-cock, M, to the bottom of the inner container and up through the absorbent material. The steam and vapours pass through N and condenser, O, and are collected in a graduated separating funnel clamped to the stand under O. P is the inlet for the cooling water and Q its overflow. The charging hole, T, through which the material is put into the apparatus, is closed by a screw-cap. Any water condensing in the vessel or surplus oil is drawn off through drain-cock F.

The steam distillation, including heating up, takes from 1 hour to 1½ hours, and is usually carried on until the absorbed naphthalene is distilled over. To avoid blockages in the condenser it is advisable to allow the cooling water to get hot before the naphthalene stage is reached.

By drawing off the hot water and running cold water through the jacket the apparatus cools rapidly and is then ready for scrubbing again; so that the complete cycle of operations can be carried out within 24 hours and therefore daily tests can be made.

According to the vapour contents of the gas being tested, from 100 to 200 c.c. of steam distillate is obtained. This is separated from the water, measured, and further examined by distillation. In most cases the estimation of benzene and toluene is the principal object. For this purpose H. G. Colman's method of fractionation and final distillation of mixtures with pure benzene, toluene, or xylene is followed.

Apart from the estimation of benzene and toluene considerable interest attaches to the examination of the lower and higher fractions. Even one day's product yields a fair amount of material for the closer examination of the extract from the gas, and a week's supply will suffice for quite considerable research.

It should be remembered that the term "benzol" in this connection refers to a mixture of a number of hydrocarbons and sulphur compounds, and may include paraffins, di-olefines, and naphthalene. Whilst at the moment the production of pure benzene and toluene is in the focus of interest on account of the supply of raw materials for explosives and dyestuffs, the identification and quantitative relation of the other components of the mixture of vapours in coal gas is of the highest importance.

The extraction of condensible hydrocarbons from coal gas has probably come to stay, and will form a valuable source of raw materials for chemicals and motor fuel purposes. As long as the present policy of gas undertakings—of supplying a more or less considerable percentage of inert gases with their product—continues, this deadweight must be compensated by enriching the gas with compounds of greater molecular weight and higher carbon: hydrogen ratio, and consequently higher calorific and illuminating value.

By a study of the compounds of this kind "naturally" present in the gas when leaving the retorts—or the superheater in the case of water gas—it will be found which of them are, economically, the most and which the least important. It will probably pay to extract the gas exhaustively, and re-incorporate with it the hydrocarbons having a lower value for other purposes; or else replace the extract or part of it by cheaper-grade petroleum distillates or cracked spirits.

However, signs are not wanting which point to the possibility of obtaining high makes of gas from coal without the necessity of dilution with nitrogen or carbon dioxide, in the not far distant future. If this comes to pass it will be possible to extract all condensable compounds from the gas and still supply a commodity which in calorific value is far superior to that of the present day.

But even now the detailed examination of the condensible hydrocarbons, beyond their customary

inclusion in "CaHm" or "illuminants," of which they form the smaller portion, is essential.

It should be realised that the value of coal gas as a commodity depends in the first instance on its chemical composition. The expression of the aggregate of its constituents and their properties, merely by a composite physical value such as the heating or illuminating power, may be convenient and necessary for the business relations of gas works with their consumers. But, for the study of the mechanism of carbonization and after-treatment of the gas and the wider aspects of the fuel problem a more extensive application of analysis to coal gas, and particularly to its smaller but not less important constituents, is highly necessary.

It is hoped that this method will be found useful in this connection. In the control of benzol extraction plants it has been found capable of discriminating between the hydrocarbons present before and after scrubbing. As is only to be expected it has been found that a large proportion of the naphtha scrubbed out from the gas consists of higher fractions which are of less immediate importance than benzene and toluene, which are frequently allowed to go forward with the gas. The aforesaid control of the extracting efficiency and a closer study of oils and solutions (benzolised oils) at the temperatures concerned is highly desirable.

It should be added that conversely the method is adaptable for the comparative testing of various oils against gases carrying known percentages of vapours both as regards their absorbing capacities and their behaviour on steam distillation.

In conclusion I wish to express my thanks to the Ministry of Munitions for their permission to publish this paper, and to Messrs. Alexander Wright & Co., Ltd., the makers of the apparatus.

## THE VULCANISATION OF RUBBER BY AGENTS OTHER THAN SULPHUR.\*

BY HENRY P. STEVENS, M.A., F.I.C.

A remarkable series of papers has recently been published by I. F. Ostromyslenski,† in certain of which he claims to have substituted successfully certain nitro compounds and peroxides for sulphur in the vulcanising process. The work, however, is of a superficial character, regarded from the standpoint of the rubber technologist. The proportions are given of a number of mixes containing the above-mentioned substances. These were cured in an autoclave or steam press at various pressures or varying periods. The vulcanisate is variously described as incompletely, completely, or over vulcanised, but there is little or no indication of the physical properties of the product, nor are figures given which would enable a comparison to be

drawn between the physical properties of the rubber vulcanised with these agents and that vulcanised with sulphur in the ordinary manner. Should it be possible to obtain results with nitro compounds or peroxides of the same order as those obtainable with sulphur, we have to deal with a discovery not only of scientific interest but possibly of great technical importance and worthy to rank with those of Goodyear and Parkes.

The first attempts to obtain vulcanisation with nitrobenzenes were unsuccessful. I was unable to obtain any indication of vulcanisation using a mixture of plantation crepe with 2 per cent. of dinitrobenzene (Le Caoutchouc et la Gutta-Percha, May 15, 1916, p. 880), and similar experiments by B. D. Porritt (this Journal, 1916, p. 988), using 1 methyl-2,4,6-trinitrobenzene, showed that this substance acts neither as a vulcaniser nor as an accelerator. However, I found later that a very considerable degree of vulcanisation can be obtained under suitable conditions and, having regard to the importance of the subject, I propose to describe in this preliminary paper some of the more important experiments which I have made.

**Vulcanisation with nitrobenzenes.** The failure to obtain positive results with nitrobenzenes appears to be due, to some extent at least, to the fact that complete translations of the original Russian were not available (*loc. cit.*). Ostromyslenski claims that complete vulcanisation can be obtained with 0.5% of nitrobenzene whereas 6% of sulphur would be required. He also states that metallic oxides, especially litharge, facilitate the process. But in the actual details of his vulcanisation experiments he only quotes two in which rubber was compounded with nitrobenzenes (in these cases 1,3,5-trinitrobenzene) without the addition of litharge or magnesia. In the first of these experiments crepe rubber was compounded with 0.8% of trinitrobenzene and he states that he obtained "complete" vulcanisation. In the second "Peruvian" with 20% of trinitrobenzene and 12% of linseed oil were heated together but vulcanisation did not take place. It is therefore plain that the evidence brought forward by Ostromyslenski that "vulcanisation" can be obtained with mixes of rubber and nitrobenzenes alone is of a contradictory nature. I have repeated the first experiment using the proportions given but with negative results, nor did the addition of 0.8% of "accelerene" (*p*-nitroso-dimethylaniline) help to produce a "vulcanised" product. Having regard to Porritt's failure and my own to obtain vulcanisation under these conditions, Ostromyslenski's claim cannot at present be accepted.

On the other hand I have obtained the effect of vulcanisation with mixes of rubber with di- or trinitrobenzene in the presence of bases such as litharge or magnesia without the least difficulty. In agreement with Ostromyslenski's statements I find that trinitrobenzene is far more efficient than dinitrobenzene. With mononitrobenzene I have not so far been able to obtain a satisfactory result.

The figures given in Table 1 were obtained with 100 parts pale sheet rubber, 8 parts litharge, and

TABLE 1.

	(1)	(2)	(3)	(4)	(5)	(6)
Parts of vulcanising agent	4	4	1	1	4	4
Cure in minutes	5	10	60	90	90	120
Colour of vulcanised rubber	Black	Black	Brown	Brown	Black	Black
Breaking load (grms. per sq. mm.)	954	876	376	318	56	52
Final length (original = 100)	8.56	7.80	3.00	8.56	5.96	3.09
Tensile product*	82	68	38	27	3	3
Acetone extract %	2.12	2.20	1.48	1.05	1.56	1.56
Nitrogen in same, calculated on rubber, %†	0.24	0.10	0.04	0.09	0.05	0.05

\* For an explanation of this term see this Journal, 1916, p. 872.

† It must not be forgotten that the acetone extract of raw rubber usually contains a little nitrogen.

the quantities of *m*-dinitrobenzene and sym-trinitrobenzene indicated. The specimens were cured in a steam vulcaniser at a temperature of 135°C. for the periods stated.

The original raw rubber gave an acetone extract of 1.8%. Comparing this with the figures for the acetone extracts of the vulcanised rubber, the figures are similar to what would have been obtained with a rubber and sulphur mix—after allowing for the free sulphur simultaneously extracted. That is to say, the acetone soluble constituents of the raw rubber are either somewhat higher or lower than those of the "vulcanised" rubber. The figures for nitrogen indicate that very little of the nitrobenzenes survived the "vulcanisation" or remained in a condition in which they were soluble in acetone. The only exception is the sample containing 4 parts of trinitrobenzene and vulcanised for 5 minutes. Fortunately in the case of the sym-trinitrobenzene we have a delicate colour reaction by which it may be detected. With a trace of alkali this substance develops a deep red coloration. Examination of the acetone extracts showed an appreciable residue of the trinitrobenzene in (1), a trace in (2), and none in (3) and (4). This test can probably be applied colorimetrically to estimate the "uncombined" sym-trinitrobenzene, which therefore becomes a very suitable vulcanising agent for quantitative experimental work.

The results of the physical tests show that the products so far obtained possess relatively poor physical qualities. The best results were obtained with the larger proportion of trinitrobenzene, but even in this case the strength as measured by the tensile product is not much more than half that obtainable with a properly vulcanised rubber and sulphur compound.

For comparative purposes, control figures are given in Table 2, (7) and (8), for a mix composed of

mix was also made with 30 parts of magnesia which gave fairly good figures. The results are given in Table 2 and apply to mixes containing 100 parts rubber and the stated proportions of the other ingredients, cured for the stated period at 140°C.

The colour of rubber vulcanised with nitrobenzenes is brown to black (see Table 1). Viewed by transmitted light in a thin stretched sheet, it is yellow-brown and translucent or almost transparent. The dark colour is discharged by treatment with hydrochloric acid after swelling with ether, with the liberation of a trace of hydrogen sulphide, so that there is little doubt but that the colour is due to the formation of a trace of lead sulphide during vulcanisation. This points to the presence of sulphur, possibly arising from traces of sulphur compounds which always hang about a vulcaniser. The amount of lead sulphide would appear to be too small to influence appreciably the reaction with the nitrobenzene or account for the "vulcanised" properties of the vulcanisate. Moreover, litharge can be replaced by magnesia (see Table 2) and probably by other basic oxides with similar results. A comparison of (9) and (10) with (7) and (8) illustrates the effect of trinitrobenzene on an ordinary rubber and sulphur mix.

**Vulcanisation with benzoyl peroxide.** The discovery of peroxides as vulcanising agents to replace sulphur was announced by Ostromyslenski (J. Russ. Phys. Chem. Soc., 1915, 1453—1461, and 1467—1471). Of the peroxides employed, most of the experiments were made with benzoyl peroxide and I therefore chose this substance for the purpose of controlling the published results. As in the case of the nitrobenzenes, Ostromyslenski's results are of a superficial nature and the vulcanisate is only very roughly characterised by stating whether the vulcanisation was complete and whether the product was dark or transparent.

TABLE 2.

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
Vulcanising agent.										
Parts of same .....	4	4	1	1	4	4	10	10	1	1
Other ingredients.										
		Litharge.			Magnesia.				Trinitrobenzene and sulphur.	
Parts of same .....	30	30	30	30	30	30			10	10
Cure at 140° C. in minutes .....	5	60	60	75	40	60	90	105	60	90
Breaking load (grms. per sq. mm.) .....	226	104	419	359	1067	1006	1238	1590	889	1223
Final length (original=100) .....	5.16	2.74	7.3	7.13	5.42	5.36	10.14	9.36	10.89	9.7
Tensile product .....	12	3	31	26	58	54	126	149	97	119
Acetone extract % .....	2.16	2.22	2.11	—	1.58	2.63				

rubber 100 parts and sulphur 10 parts. With a view to obtaining higher figures some experiments were made with larger proportions of litharge (30 parts) but the results were less satisfactory than with the smaller proportion (3 parts) previously used. A

As is well known, benzoyl peroxide, like other peroxides, is very explosive and consequently cannot be handled in the dry state. For these experiments it was used in the form of a paste with about its own weight of water. In this condition

TABLE 3.

	(1)	(2)	(3)	(4)	(5)	(6)
	A.		B.		C.	
Cure (minutes) at 130° C. ....	60	30	60	30	15	10
Benzoyl peroxide % .....	0.33	0.33	1.33	1.33	6.67	6.67
Breaking load (grms. per sq. mm.) .....	34	29	36	57	412	338
Final length (original length=100) .....	223	181	219	339	1025	1006
Tensile product .....	8	5	8	19	42	39
Acetone extract .....	1.17	2.04	2.00	2.01	2.13	3.31



it presents no difficulty when added to the rubber on the mixing rolls and the small amount of water added is lost by the time the mixing is complete.

In the first series of experiments three mixes were made of the same plantation crepe rubber with the following percentages of benzoyl peroxide (reckoned dry):—A, 0.33%; B, 1.33%; C, 6.67%. Small portions of the sheeted rubber were cured for various periods to ascertain by simple hand tests the cure most likely to suit the particular mixing. With mixings A and B it was found impossible to prepare an even moderately strong rubber, but with mixing C a fairly satisfactory result was obtained. The figures for the tests are given in Table 3.

It will be noted that mixings A and B give very weak rubbers. Nevertheless, the influence of even 0.33% of benzoyl peroxide is clearly marked, as the raw rubber heated alone under the conditions under which these samples were cured would have given a weak, sticky mass whereas the vulcanised samples A, although very weak and adhesive between cut surfaces, were nevertheless superior in physical properties to what would have been produced had the rubber been heated without the benzoyl peroxide. Mix C gave the best results, the vulcanised samples having approximately one-third the strength of a similar rubber vulcanised with sulphur in the heat. Freshly cut surfaces are rather adhesive and the rubber has the feel of being somewhat undercured. Yet on the whole there is a very marked difference between these samples and those containing the smaller proportions of benzoyl peroxide. Samples from mix B occupy an intermediate position. All the vulcanised samples are very pale in colour, quite as pale as the original raw rubber. Those of mix C have indeed a very attractive appearance. They are quite transparent and resemble some of the palest cold cured sheet prepared from plantation crepe rubber.

The samples were acetone extracted a few days after curing and the figures for acetone extract are given in the table. These compare with an acetone extract of 3.0% for the original raw rubber.

The behaviour of the vulcanised specimens to solvents was also noted. In this respect they exhibit a gradual change in properties according to the proportion of benzoyl peroxide in the mix. Thus the vulcanised mix A "dissolves" in a few hours in benzene in a similar manner to a raw crepe rubber. Mix B swells enormously but does not "dissolve." It remains a soft, shapeless, gelatinous lump in the solvent. Vulcanised mix C behaves in the same manner as an ordinary vulcanised rubber, that is to say, it swells considerably but retains its original form. It may therefore be said that the change brought about by heating with benzoyl peroxide is exactly similar to that produced by sulphur and that the products formed have similar properties. The proportion of benzoyl peroxide required to give a fairly complete vulcanising effect is similar to the proportion of sulphur which would be required for the same purpose, but the vulcanisation with benzoyl peroxide is brought about in a much shorter time. Thus, in the above experiments and also in those published by Ostromyslenski, vulcanisation is fairly complete in ten or fifteen minutes at 130°–135°C. with 4–6% of benzoyl peroxide, whereas vulcanisation would have hardly begun with a mix containing 5–8% of sulphur and would require 2–3 hours for completion. On the other hand, attempts I have made to obtain satisfactory vulcanisation with smaller proportions of benzoyl peroxide, but vulcanising for longer periods, have not been successful. Thus it will be seen that with 1.33% of benzoyl peroxide better results were obtained when vulcanising for 30 minutes than for 60 minutes. Further experiments are in progress.

## Manchester Section.

Meeting held at Grand Hotel on Friday, December 1st 1916.

MR. J. H. ROSEASON IN THE CHAIR.

### SPONTANEOUS IGNITION TEMPERATURES OF LIQUID FUELS FOR INTERNAL COMBUSTION ENGINES.

BY HAROLD MOORE, M.Sc.TECH.

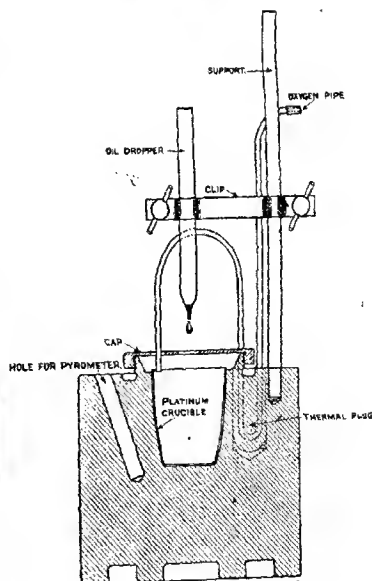
Determinations of the temperature of spontaneous ignition or ignition point of liquid fuels were first made by Holm in 1913\*, and their importance as a test is not yet fully realised.

Flash points and burning points are common tests used for both liquid fuels and lubricating oils. The flash point is the temperature at which an oil will give off sufficient vapour to burn momentarily when a small flame is held close to the surface of the oil. The burning point is the temperature at which vapour is given off by the oil at a sufficient rate to maintain a continuous flame.

These properties provide a measure of the danger from fire which is encountered in the handling and storage of liquid fuels and illuminants. They are of little use as an indication of the behaviour of a fuel in an internal combustion engine. The temperature of spontaneous ignition is the temperature at which a substance surrounded by oxygen or air at the same temperature will burst into flame without the application of any spark or other local high temperature.

Holm made several determinations of spontaneous ignition in a tube furnace whilst Constam and Schl pfer investigated the properties of a large number of fuel oils for Diesel engines in a simple apparatus consisting of a small platinum crucible

\* Z. angew. Chem., 1913, 273.





placed in a larger crucible, the intervening space being filled with sand. No means of preheating the incoming air or oxygen was employed, and the experimenters concluded that the error was  $\pm 30^\circ \text{C}$ .

Some time ago the author required to know the temperature of spontaneous ignition of several fuel oils with which he was experimenting on a Diesel engine, and as no existing apparatus was sufficiently delicate to differentiate between these oils it was determined to make a special instrument for this purpose. The result was the device shown in the accompanying figure.

The instrument consists of a diffusion block of 4 in. diameter mild steel bar  $3\frac{1}{2}$  in. deep, the base of which has been turned in order to afford a greater heating surface. The upper end of the bar has been machined so as exactly to fit the platinum crucible used for the estimation of the coking values of oils (dimensions, upper diameter 35 mm., diameter of base 22 mm., height 37 mm.).

A screwed-on cover protects the upper end of the crucible from draughts. This cover is provided with two holes, one for oxygen or air inlet and one for oil inlet. The oxygen or air is preheated to the temperature of the experiment, passing through a thin copper coil situated in a chamber inside the block.

A thermometer and a thermocouple pyrometer are fitted into the diffusion block as close as possible to the base of the platinum crucible and serve to show the temperature of the experiment.

The method of operation when determining values in oxygen is to pass the gas through the apparatus at a speed of three bubbles per second. The quantity of oxygen is observed by means of a wash bottle filled with sulphuric acid, which also serves as drying agent. When the temperature of the block is constant at a desired temperature, one drop of the fuel under examination is allowed to fall through the hole in the centre of the cover of the instrument. If the temperature is much higher than the ignition point a sharp explosion will occur almost immediately, if below the ignition point no explosion takes place. When only slightly above the temperature of ignition, the explosion follows some 10 to 12 seconds after the introduction of the oil.

By repeating the experiment the lowest temperature at which ignition takes place is rapidly determined. This temperature is the temperature of spontaneous ignition. The results obtained by different experimenters using the same instrument agree to within  $3^\circ \text{C}$ .

A similar procedure using air instead of oxygen will give the ignition temperatures in air, but in this case the explosion is much less violent and always follows one second after the introduction of the oil.

Spontaneous ignition temperatures are of importance for several purposes, but in this paper I will only deal with the application of this test in the examination of liquid fuels for use in internal combustion engines, in which respect I consider that they are of as much importance as the heat value.

Internal combustion engines, whether operating on 2 or 4 stroke cycles, may be divided into two classes:—

1. Engines which receive the fuel and air before the compression stroke, *e.g.*, petrol and gas engines.
2. Engines which compress the air alone and inject the fuel into the cylinder about the end of compression, that is immediately before inner dead centre, *e.g.*, Diesel engines.

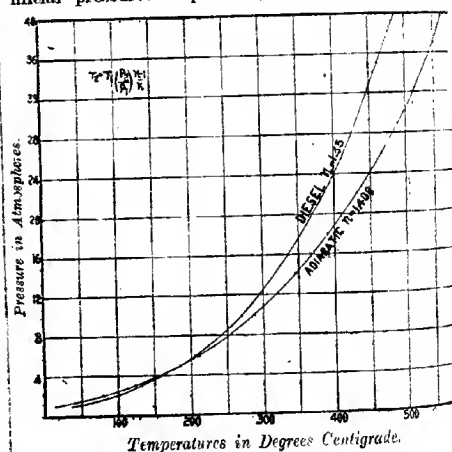
For the first type of engine it is generally desirable to employ high compressions in order to obtain high thermal efficiencies, but the compression allowable is limited by the liability to pre-ignition. Pre-ignition is most liable to occur when the temperature of the mixture due to the heat of

the cylinder walls and the heat of compression approaches the ignition point. Pre-ignition is accompanied by fall of power and loss in efficiency.

In Diesel engines there is no danger of pre-ignition as no fuel is present in the cylinder at the time of compression. No special means are adopted for igniting the charge in Diesel engines, firing being dependent upon the spontaneous ignition of the fuel immediately it enters the cylinder. In Diesel engines it is therefore necessary to employ high compression pressures in order to obtain a sufficiently high compression temperature to ensure the spontaneous ignition of the charge. Provided the temperature is attained it is generally advisable to keep the compression pressure low for mechanical reasons. From these facts it is seen that both for engines which receive fuel and air before the compression stroke and for Diesel engines the temperature at which the fuel will ignite spontaneously is of fundamental importance. In the first case, one is forced to employ a low compression pressure on account of danger of spontaneous ignition, whereas in the case of the Diesel engine this limitation does not appear, but a high compression is required in order to exceed the ignition temperature of the fuel. The temperature attained in the cylinder of an internal combustion engine during compression can be calculated by means of the formula—

$$T_2 = T_1 \left( \frac{P_2}{P_1} \right)^{\frac{n-1}{n}}$$

where  $T_2$  and  $T_1$  are respectively final and initial temperatures absolute, and  $P_2$  and  $P_1$  are final and initial pressures respectively;  $n$  is a constant



Press. in atmos.	Temp. abs.	Adiabatic temp. °C.	Diesel temp. °C.
1	288.0	15.0	38.0
2	352.0	79.1	108.0
3	398.0	123.0	133.0
4	430.0	157.0	162.0
6	459.0	186.0	185.0
8	485.0	212.0	207.0
10	506.0	233.0	224.0
12	—	—	—
14	619.0	346.0	336.0
16	—	—	—
18	664.2	391.0	379.0
20	—	—	—
22	705.2	432.0	—
24	739.0	466.0	426.0
26	—	—	—
28	780.1	513.0	447.0
32	—	—	—
40	490.5	567.0	497.0

which for air under adiabatic conditions equals 1.408.

On account of the leakage of gas past the piston and the cooling due to the cylinder walls the constant,  $n$ , in internal combustion engines approximates to 1.35, the conditions being between those of isothermal and adiabatic compression.

By means of this formula it is possible to calculate approximately the temperature which corresponds to any given compression. The graph of pressure-temperature here shown has been calculated by this means.

It is not at present possible to determine the temperature of spontaneous ignition under actual cylinder conditions, but the relative value can be accurately determined by the device described above. The influence of pressure upon the ignition temperature is a subject for further research.

By an experience of the difference between compressions which have been calculated from the determination of the spontaneous ignition in the instrument, and the experimentally determined point at which spontaneous ignition takes place in an engine, it is possible to build up tables showing the approximate relationship between the experimentally determined ignition point and the maximum compression practicable in a petrol engine. By a similar process it is possible to ascertain the minimum pressures which can be employed in a Diesel engine with any given fuel.

As an example of the influence of the ignition temperature of a liquid fuel we may take the case of alcohol. It has been found in practice that alcohol, though much lower than petrol in calorific power, when run in an engine can be made to yield approximately the same power per unit volume as the latter fuel.

The net calorific power of petrol is about 10,450 calories per gram or 7315 cal. per c.c. The net calorific power of commercial alcohol is about 5420 calories per gram or 4440 cal. per c.c. If both be burnt in an engine with the normal compression (i.e., compression adjusted to suit petrol) the consumptions per B.H.P. hour will be approximately in inverse proportion to the calorific powers of the fuels and over 1½ galls. of alcohol is required to do the same work as one gall. of petrol. Now the spontaneous ignition temperature of petrol (in oxygen) is about 272° C., whilst that of commercial alcohol is 395° C., therefore alcohol will withstand a much higher compression.

The compression pressure of a petrol engine tuned to run on petrol is approximately 90 lb. per sq. in., but with alcohol this pressure may be raised to 200 lb. per sq. in. and by this means the overall thermal efficiency of the engine is raised from about 22% to 35%, when it is found that the volumetric consumption of alcohol per brake horse power hour is approximately the same as that of petrol. Thus though petrol possesses 65% greater calorific power than alcohol per unit volume, the advantages of this high heat value are entirely lost on account of its low ignition point. The ignition temperature is of general interest to chemists as it is a measure of the relative stability of the bodies towards heat. The accompanying table shows the spontaneous ignition temperatures of several fuels which have been determined with this instrument.

#### Temperatures of Spontaneous Ignition.

Lab. No.	Description.	Specific gravity.	Spontaneous ignition temp. in oxygen.	Spontaneous ignition temp. in air.	Difference.
521	Petroleum Distillates, Pratt's Perfection Spt. No. 1	0.710	272	383	+111
256	Petrol (Mex.)	0.718	279	361	+82
522	Pratt's Spt. No. 2	0.724	270	371	+101

Lab. No.	Description.	Specific gravity.	Spontaneous ignition temp. in oxygen.	Spontaneous ignition temp. in air.	Difference.
			°C.	°C.	°C.
520	Petroleum Distillates, Taxibus Spt. (A.A.O. Co., Ltd.)	0.729	272	390	+118
26	Paraffin oil from A.A.O. Co.	0.807	251	—	—
350	Petrolite Kerosene Empire Paraffin	0.814	251.5	432	+180.5
	Petrol from Anglo-American	0.782	253	395	+142
	Lamp oil from Anglo-American	0.785	—	392	—
Gas oil.	Gas oil (A.A.O. Co., Ltd.)	0.787	—	367	—
	Petroleum (crude and residue).	—	254	458	+104
118	Crude Petroleum (Egypt)	0.851	260	—	—
121	Dibhol oil (Assam)	0.890	261	384	+123
124	Anglo-Persian Oil Co.'s Oil	0.894	254	408	+154
131	Crude Petroleum (Texas)	0.895	256	387	+131
8	Anglo-American Fuel Oil	0.900	269	430	+161
168	Anglo-Mexican Oil	0.908	259.5	417	+157.5
32	Crude Petroleum (Texas)	0.936	268.5	416	+147.5
120	Crude Petroleum (Borneo)	0.939	269	380	+111
111	Mexican Fuel Oil	0.948	259.5	424	+164.5
31	Crude Petroleum (Mexican)	0.949	253	425	+172
47	Crude Petroleum (Trinidad)	0.950	274	424	+150
61	Crude Petroleum (California)	0.952	264	—	—
167	Venezuelan Petroleum	0.955	275	420	+145
13	Crude Petroleum (California)	0.961	262	420	+158
	Shale Oils.				
513	Oil Engine Oil (Broxburn Oil Co., Ltd.)	0.768	233	333	+80
318	Lighthouse Oil (Broxburn Oil Co., Ltd.)	0.803	251	322	+71
	Tar Distillates.				
	Xylol Commercial	0.860	484	—	—
237	Tolnol 90%	0.863	518	—	—
140	Benzol 100%	0.875	566	—	—
	Premier Tarless Tar Oil	0.992	340	—	—
361	Creosote Oil (Hardman & Holden)	1.010	415	—	—
58	Water Gas Tar (Creosote (Stainsby & Lyons))	1.036	473	—	—
71	Coke Oven Tar Oil (Simon Carves)	1.046	478	—	—
262	Tar (Product of low temp. carbonisation)	0.987	307	508	+201
371	C.W.G. Tar (Stockport Gas Works)	1.074	464	—	—
340	Oil Gas Tar (Beckton)	1.077	415	—	—
346	Horizontal Retort Tar (Heywood Gas Works)	1.114	445	—	—
355	Horizontal Retort Tar (Stockport Gas Works)	1.123	451	—	—
342	Coke Oven Tar (Simon Carves)	1.132	494	—	—
363	Coke Oven Tar (Copper Co.)	1.140	488	—	—
362	Coke Oven Tar (Koppers Type Ovens)	1.145	495	—	—
267	Blast Furnace Tar (Carlton Iron Works)	1.172	498	—	—
274	Blast Furnace Tar (Wm. Baird & Co.)	—	410	—	—
	Miscellaneous.				
142	Alcohol	0.817	395	518	+123
	Turpentine	0.842	275	275	same
305L	"Mirreco-Diesel" Compressor Lubricating Oil	0.875	265.5	405	+139.5
249L	"Mirreco-Diesel" Engine Lubricating Oil	0.894	265.5	401.0	+135.5

Lab. No.	Description.	Specific gravity.	Spontaneous ignition temp in oxygen.	Spontaneous ignition temp in air.	Difference.
335	Miscellaneous.		°C.	°C.	°C.
	Whale Oil.....	0.921	273	470	+197
	Ether.....	0.730	190	347	+157
	Paraffin Wax.....	—	245	—	—
B26	Naphthalene.....	—	402	—	—
	Free Carbon from Tar.....	—	348	—	—
	Asphaltum from Oil.....	—	290	—	—

From general observations the author has concluded that:—

1. Compounds containing simple molecules have higher ignition points than similar compounds containing more complex molecules. This rule applies to all types of compounds.
2. The ignition points of aromatic compounds are much higher than those of aliphatic compounds.
3. Unsaturated hydrocarbons have lower ignition points than the corresponding saturated hydrocarbons.
4. Ignition points observed in air are higher than those observed in oxygen. This difference for petroleum products is generally 100° to 200°C.

#### DISCUSSION.

The CHAIRMAN observed that the percentage of hydrogen in the compounds appeared to have some effect upon the lowering of the ignition point.

Mr. J. DRUMMOND PATON said from investigations he had conducted in the United States with regard to gas producers for turbines for marine purposes it was discovered that the determining factor causing rapid ignition and pre-firing was the hydrogen content. It was then endeavoured to ascertain the time constant for ignition of fuel of various hydrogen contents and from the velocity of ignition the actual value was set. It was possible that according to the nature of the compound and the molecular tenacity of the hydrogen the tendency to free liberation would be the determining point, and this determined the linear velocity of ignition.

Mr. L. E. VILES remarked that the spontaneous ignition point of petrol was much lower than that of benzene. Motor car users were aware that even with the same compression better effect could be obtained from benzol than from petrol. He asked whether designers of explosion engines had taken this fact fully into account by producing an engine to run with benzol, which according to the evidence Mr. Moore had brought forward, should be more efficient than an engine indiscriminately used either with benzol or petrol.

Mr. DRUMMOND PATON said the most important heat loss occurred in the period of ignition. Had any investigation been made with a view to isolating the crown of the engine, so that at the actual ignition, the cooling effect and surface of the cylinder would be a minimum? For instance, cast iron was now used. Was it feasible to cover the crown and piston face with isolating material which would diminish heat loss?

Mr. T. O. MORGAN, speaking from the point of view of a motorist, said he had tried various mixtures of petrol and very light petroleum, and had even gone so far as to experiment with mixtures of naphthalene and benzol, without noticing any material difference in the pre-ignition or the overheating of the engine.

Mr. MOORE, in reply, said it was very noticeable that compounds with a high hydrogen content usually had a low spontaneous ignition temperature, but the rule did not apply in several cases, whereas the rules given at the end of the paper could always be applied. Petrol contained more

hydrogen than kerosene, but petrol possessed the higher ignition temperature. Paraffin wax had a still lower hydrogen content, but possessed the lowest ignition point on account of its complexity. Methane had a very high hydrogen content and high ignition temperature. It would be seen that hydrogen content was not a reliable indication of the ignition temperature. It was well known that benzol would stand a much higher compression pressure than petrol. The engine of a racing motor car running at 100 lb. compression would "knock" very badly on petrol. It would work much better on benzol. At the same time it was possible to have pre-ignition with any fuel at comparatively low compressions. There were two causes for pre-ignition. If the temperature of the engine were the same at all parts there would not be pre-ignition until the temperature of compression corresponded with the ignition temperature. If the engine were not perfectly even in temperature, as must always be the case in practice, a plug point or a valve top would get hot and act as a firing agent. This ignition was quite independent of spontaneous ignition, so that an engine might "knock" without the compression being excessively high. The adiabatic curve was calculated from the adiabatic formula, the formula of the other curve (for Diesel engine) being taken from the tangent on the compression curve of the indicator diagram of a Diesel engine. Mixtures with naphthalene had, of course, been largely used. The German army was now running its motor vehicles on mixtures of about 4 volumes of alcohol with one volume of a solution of naphthalene in benzol. The addition of naphthalene cheapened the mixture. The benzol raised the percentage of carbon in the mixture and increased the calorific power. Replying to a further question, Mr. Moore said that there was a tendency for the spontaneous ignition temperature to vary inversely with the flash point, but these values were better considered as independent.

#### Newcastle Section.

Meeting held at Bolbec Hall on Monday,  
January 17th, 1917.

MR. HENRY PEILE IN THE CHAIR.

#### UTILIZATION OF WASTE HEAT FROM COKE MAKING.

BY HENRY PEILE.

Up to comparatively recent years very little of the waste heat from coke making was utilized except for the purpose of raising steam for colliery purposes. This was usually done by installing Cornish or Lancashire boilers on the flues carrying the waste heat from the beehive ovens to the chimney. In this way only a small portion of the waste heat was utilized, only one lb. of steam per lb. of coal carbonized.

In 1904 when the patent ovens were erected at Blaydon there was added a complete installation of water tube boilers which are fired by the waste heat and gas generated. The steam from 80 ovens is carried direct into an up-to-date power station. The whole of the power generated at this station is delivered into the mains of the Newcastle Electric Supply Company for general distribution in the Newcastle district. In this way the whole of our waste heat and waste gas is utilized to the best advantage as the station is kept running day and night.

At Rowlands Gill there is an installation of 200

beehive ovens. About eight years ago it was decided to utilize the waste heat from these ovens for the production of electricity. Nine Stirling water tube boilers were erected on the main flues which carry the waste heat from the ovens to the chimneys, and where it was found necessary steam driven fans were added to increase the draught on the ovens.

Economizers were also fitted at the back of the boilers so as to avoid any loss of waste heat. As the process of making coke in beehive ovens is more or less intermittent it was decided to erect two Bettington tubular boilers which are fired with a mixture of coal and coke ballast. The fuel is elevated into an overhead bunker from which it falls into a disintegrator where it is ground into a very fine powder. This powder is then blown into the boiler by hot air where it burns with a hot flame, combustion being very complete. Each boiler is capable of producing 15,000 to 20,000 lb. of steam an hour.

In addition to these two boilers a battery of four Stirling boilers was erected, fitted with underfired movable grates specially designed to burn ballast, one of the waste products from coke making. Burning coke ballast is one of the most economical methods of raising steam and at the same time getting rid of waste material from ovens which has otherwise to be disposed of at some cost. This ballast contains about 80% carbon and is therefore a very valuable fuel. From these six boilers it is possible to add to the steam from the coke ovens 60,000 lb. of steam an hour. The whole of the steam from these boilers is superheated 200° C., and carried direct into the power station.

In the Whinfield Power Station at Rowlands Gill there are installed four Westinghouse turbines fitted with three phase alternators working at a voltage of 6000. All the semachines are interchangeable.

The electricity which is generated at 6000 volts is passed through specially designed transformers, being supplied to the Newcastle Alloy Company at varying voltages suitable for use in their electric furnaces.

As the station is run on a continuous load night and day it is found necessary to keep the transformers cool by means of oil and water. The output of the station approaches thirty million units a year. Special transformers are installed for supplying power to the colliery for pumping, hauling, and lighting purposes.

Electricity is also supplied for lighting purposes in the district.

#### *Ferro-alloys.*

The low tension current from the transformers is passed through the walls of the power station into the Alloy Company's works where it is utilised for the manufacture of ferro-alloys and other purposes.

In the early stages of the company experiments for the manufacture of ferro-silicon were carried out in a small electric furnace. On the strength of these experiments larger furnaces were erected until to-day there is a 2000 K.W. furnace producing from 10 to 15 tons of ferro-silicon a day. During the last four or five years the following grades of ferro-silicon have been successfully produced: 25%, 50%, 75%, and also ferro-silicon of exceptional purity, containing 95% of silicon. There is at present under consideration a scheme for the production of one to two thousand tons a year of this material for war purposes.

We have also produced carborundum in our furnaces, but up to now this has not been done on a large scale.

Before the war there was a furnace running on carbide which made one or two thousand tons of

this material, but owing to keen competition it was decided not to continue its manufacture.

The next alloy to be taken in hand was ferro-molybdenum. Considerable difficulty was experienced in making this alloy on account of its very high melting point and the difficulty of getting rid of objectionable impurities. These difficulties have been successfully overcome and an alloy of excellent quality, testing over 80% of molybdenum, is now being produced.

At the beginning of the war it was discovered that practically there were no stocks of ferro-chrome in the country. We were asked to make this alloy and within three or four weeks of war breaking out we were in a position to supply our customers. Since then we have made large quantities of all grades of ferro-chrome with carbon content ranging from 1% to 10%. The 1% carbon grade has a very fine crystalline fracture.

Two or three months after war breaking out a large demand for ferro-tungsten set in for the manufacture of high speed steel for munition works. To make this material we erected a specially constructed furnace house fitted with furnaces suitable for the manufacture of this alloy. We are now producing between 300 and 400 tons of ferro-tungsten a year, all of which is being supplied to steel makers in Sheffield. The alloy is sent out in a ground state or in small pieces as required. Considerable difficulty was experienced at first in breaking up this alloy. These difficulties have now been overcome.

Cuprous oxide. Before the war this oxide was all made electrically in Germany. At the present time we are producing between 300 and 400 tons a year.

Our objects in putting up these works were first to utilize the waste heat from our coking plant and second the production in this country of the various ferro-alloys which were all being supplied from abroad. The value of this policy has been fully justified since war broke out, as otherwise great difficulty would have been experienced in obtaining the necessary supplies of alloys to carry on munition work.

I think that perhaps I have said enough to show how a waste product from coke making can be turned to good account either in producing electricity for general purposes or for the production of alloys and for use in other processes in which electricity is employed. If we are to meet competition in the future, we must be prepared to see that there is no waste of any sort going on in our works.

#### **Yorkshire Section.**

*Meeting held at Queen's Hotel, Leeds, on Monday, January 8th, 1917.*

PROF. J. W. COBB IN THE CHAIR.

#### **NOTE ON A DANGEROUS OIL USED IN A WOOLLEN MILL.**

BY THOMAS FAIRLEY, F.I.C., AND R. A. BURRELL, F.I.C.

The oil which is the subject of the present note was being used as a wool oil by a firm manufacturing woollen goods. During the process of manufacture, it was observed that white wool when oiled in the usual manner rapidly began to

heat and would no doubt have eventually fired if it had been left unattended to. It was therefore desirable to determine the nature of the oil and its consequent risk of fire.

The oil gave the following numbers:—

	°C
Free fatty acids (as oleic acid) .....	87.6
Unsaponifiable matter .....	1.0
Cotton seed oil .....	none
Iodine value .....	138.0
Yield of insoluble bromides (m.p. above 200° C.) .....	40.1
Saponification value .....	193.6
Yield of fatty acids by saponifying, decomposing the soap and drying at 100° C. ....	98.2
Iodine value of separated acids .....	137.0
Loss on heating for 1 hour at 340° F. ....	66.0
Flash point (open test), 368° F. ....	—
Ignition point, 400° F. ....	—

The oil therefore consisted of free fatty acids admixed with about 12% of neutral oil. It had a decided fishy smell and its fish origin was further confirmed by the yield of insoluble bromides and their behaviour on heating, as they did not melt at 200° C., at which temperature they began to blacken and decompose, whereas the hexabromides from linseed oil melt to a clear liquid at about 170° C.

The iodine value and the loss experienced on heating showed the oil to be quite unsuitable for use in a woollen mill and this deduction was completely confirmed in actual practice, as the use of this oil nearly involved a serious fire.

We have examined a large number of oils for the Insurance Companies but have not met with a similar oil.

#### DISCUSSION.

Mr. W. Mc.D. MACKEY was surprised to find so much of the oil volatile at 340° F. He would like to know the composition of the distillate, and whether any water was present. He thought the use of such an oil would make the yarn sticky.

Mr. J. K. S. DIXON said there were large quantities of fatty acids of all kinds on the market at present, among them fish oil fatty acids. The glycerin was required for explosives, and the resulting fatty acids were being used for a variety of purposes, but this was the first time he had heard of fish oil fatty acids being used (except as an adulterant) as a wool oil, for they were undoubtedly dangerous. He was not altogether surprised at the loss on heating at 340° F. In his experience it was almost impossible to obtain a constant weight when drying fatty acids of any kind at a temperature above 110° C.

Mr. B. T. VICKERS did not think the iodine value a reliable criterion as to whether the oil would heat. It only showed the total possible absorption of oxygen and gave no indication of the rapidity with which oxygen was absorbed, which was the real cause of heating. An actual test in a Mackey's tester would be more satisfactory. He thought it possible that this oil might produce "gilding" which was quite different to heating.

Mr. J. F. BARNES said that in his opinion many cases of spontaneous heating were caused by the melting of oil with live steam in the casks; when the resulting oil and water were put on the wool it was more liable to heat than the oil by itself.

Mr. WM. RUSHBY said that it was a common practice in the United States to use emulsions made of lard oil and water with a little borax. Hence it was evidently not a danger to have water present with oil on the wool-fibre.

Mr. BURRELL replied that the loss on heating was certainly abnormal; it was not due to the presence of water. He regretted that the small quantity of oil placed at their disposal did not permit of an investigation into the nature of the fatty acids present. He did not think, however, that there could be any notable proportion of either linolic or linolenic acids as the insoluble bromides from these acids melted about 175° C., whereas those obtained did not melt at 200° C., and on further raising the temperature began to decompose.

The oil was not distilled but heated in a flat-bottomed basin in an air oven kept at 340° F. This temperature had been selected because it was the minimum flash point allowed by the Insurance Companies for oils used in woollen mills. The loss of weight took place in a non-oxidising atmosphere, viz., the oil's own vapour; when a free current of air was passed through the oven it was not so great, as it was counterbalanced by the gain of oxygen in the residuum, which assumed a hard, varnish-like consistency. Oleic acid was usually regarded as a non-volatile substance and required a temperature of about 482° F. (250° C.) to distil in a current of superheated steam, but when heated in an air bath at 340° F. it lost about one-third of its weight. Some of the cloth oils which contained from 30%—50% of free fatty acids would lose at least a quarter of their weight when heated under similar conditions. He agreed with Mr. Dixon that it was very difficult to attain a constant weight when heating free fatty acids. A good illustration was the washed insoluble fatty acids from butter when dried at as low a temperature as 100° C.

The iodine value was of great use in determining the safety or otherwise of wool oils; it was the measure of the capacity of the oil for absorbing oxygen and therefore of its drying tendencies, and Gellatly had shown the intimate relationship which exists between drying oils and their tendency to spontaneous combustion when exposed to the air in a finely divided state, as when spread on wool. Woollen manufacturers rightly objected to such oils, as not only was there the tendency to spontaneous combustion but they spoiled the yarn by "gunning." With this oil the wool had not merely "gilded" but had actually charred and the mill itself might be regarded as the cloth oil tester.

In this country the use of emulsions or "creams" was still in its infancy, but no doubt the presence of water along with the oil would tend to increase the heating effect. On the other hand their experience of emulsions was that they were generally made from a safe non-drying oil such as olive oil.

#### NOTES ON THE WORKING OF A DUST FIRED BOILER.

BY C. A. KING, M.S.C.

During recent years much discussion has taken place on the necessity for improved utilisation of fuel of every grade, and with this in view it may be of interest to note a recent appliance, which makes use of low-grade raw coal for steam-raising purposes.

It is well known that in ordinary boiler practice the coal lies on the grate in a fairly thick bed with irregular contact with air, so that immediately after firing, the supply of air is insufficient, resulting in incomplete combustion and the production of smoke; and later, when the volatile matter of

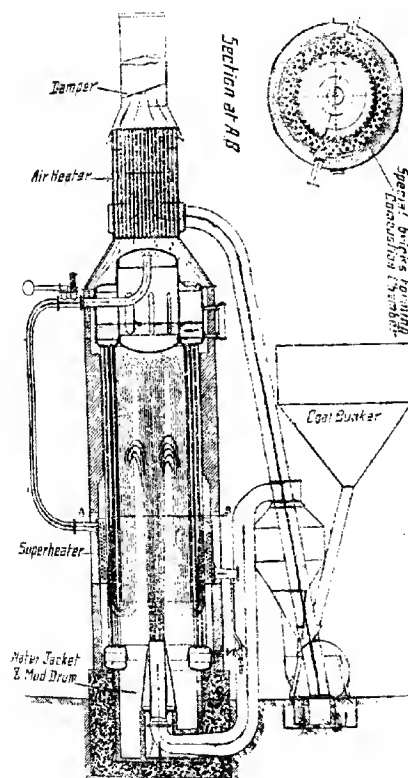
the coal has been distilled off, excess of air passes through the flue, both conditions giving rise to considerable loss of useful heat.

On the other hand, with gaseous firing, the combustible matter, in the form of gas, comes into intimate contact with the air supply and is fully burned without the necessity of a large excess of air to ensure complete combustion.

The method of firing the type of boiler to be described can be regarded as being intermediate between these two conditions, the better admixture of fuel and air being secured by grinding the coal to a very fine dust and blowing the required mixture of coal-dust and air together into the combustion chamber of the boiler. It will readily be seen that the particles of coal are brought into much more intimate contact with air than in standard steam boiler practice.

Commercially and scientifically it is important that fine coal, which is a positive source of danger when left in mines, and is unsaleable at the present time by reason of its high ash content, and its lying too closely on boiler grates and in gas producers, can be successfully used.

The Battington boiler (see fig.) is an example of



this type. Small coal is fed from a storage hopper by means of a variable worm gear, into a pulveriser which also acts as a fan, drawing heated air for the combustion of the coal from a

tubular air heater above the boiler. The pulverised coal, carried along by the air current through a separating chamber, across which is stretched a sieve to reject the coarser particles, is burned at the mouth of a water-cooled vertical nozzle. The heated air, in addition to regenerating some heat from the flue gases, to a certain extent dries the coal as it passes through the pulveriser, preventing clogging of the screen.

The dust as used is of such a fineness that 80% of it will pass through a sieve of 200 meshes per linear inch.

The boiler proper consists of concentric vertical rows of tubes terminating at the lower end in an annular metal chamber, and at the upper end in a drum, the combustion chamber being formed by fire bricks of semicircular shape lining the innermost ring of tubes.

The flame and hot gases travel vertically up the central axis of the boiler until, nearing the drum, they are turned down and under the firebrick wall, passing again vertically amongst the water, superheater, and air-heater tubes, and finally to the chimney. It should be noted that in the vertical design the action of gravity causes the coal-dust to travel the combustion chamber a second time, whereas in a horizontal or slightly inclined installation the dust tends to fall out of the combustion zone under this influence.

The function of the firebrick lining, apart from acting as a boundary for the fused ash pellicles, is to retain a sufficient quantity of heat to re-ignite the fuel mixture after any temporary stoppage. Its use also prevents short circuiting of the gases around the tubes, and thus gives an increased length of contact for the combustion process to take place. The incandescent surface of the brickwork may also tend in the direction of more complete combustion.

The ash of the fuel in its passage through the furnace of the boiler is converted into a semi-liquid spray, which coalesces on the lining, slowly trickles down, and drips from the bottom edge into the ashpit, from which it is raked out at intervals. It is of obvious advantage to use a coal the ash of which has a low fusion point, or to modify a refractory ash by suitable addition to the coal. Many analyses show that this clinker contains no combustible matter, and theoretically this is excellent, but with furnaces of this type it is usual to find in practice that a certain proportion of the coal is not completely burnt to ash but passes out of the boiler as a coked dust containing up to 40% of fixed carbon and sometimes up to 5% of volatile matter. This is undoubtedly only a question of design, but at the present time a dust catcher may sometimes be usefully installed.

The ultimate test of the efficiency of a boiler, however, is its evaporative power. It has long been the custom to compare boilers upon the basis of the number of pounds of water evaporated by each, per pound of fuel, but this is likely to be deceptive, especially if, as seems probable, coals of much inferior quality are brought into use. The true efficiency of a boiler is represented by the number of heat units which are utilised in heating the water and converting it into steam compared with the total number of heat units supplied in the fuel used. In the results of the tests below both methods of expression are given.

On one day two tests were made. No interruption of the ordinary working condition was made except that the coal was weighed and the water measured. Temperatures and pressures at various points were also taken throughout the test. The boiler was run at about normal load for 5½ hours

(Test I), and then for two hours with 30% overload (Test II).

Pressures.				Temperatures ° C.						Flue gas.					Fuel.		Fuel supply.	Amps. to motor 44 C.V.				
Time.	Damp. open sq. in.	Fuel pipe air enlg. cm.	Steam lb.	F. gas leaving air heater.	F. gas enlg. air heater.	Air enlg. heater.	Hot air.	Fuel mixt.	Feed water.	Sadd. steam.	Super-heated steam.	Water twyers.	% CO <sub>2</sub> .	% O <sub>2</sub> .	Sampled from.	% loss in F. gas.	Analysis.		Fuel.	Alt.		
																	Moist. matr.	Ash.				
8.30	198	8.75	160	235	235	—	158	63	12.5	175	—	—	—	—	—	—	—	1.6	22.7	13.8	—	—
9.0	198	8.75	160	235	235	—	165	64	25.5	180	295	—	11.8	0.2	L.A.H.	—	—	Cal. val = 115,000 B.T.U.	—	—	—	—
9.30	198	10	160	307	307	—	180	78	39	180	290	—	11.8	0.2	L.A.H.	—	—	—	—	—	—	—
10.30	198	10	160	312	312	—	185	76	46	181	297	—	9.2	0.0	L.A.H.	—	—	0.0	100.0	—	—	—
11.30	198	8.75	160	312	312	40	185	76	46	182	300	—	9.4	12.0	L.A.H.	—	—	—	—	—	—	—
11.40	198	10	160	318	290	41	170	63	52	180	297	—	11.2	7.0	L.A.H.	—	—	—	—	—	—	—
12.30	198	10	160	318	298	41	177	62	53	180	290	—	12.0	6.8	L.A.H.	—	—	—	—	—	—	—
1 p.m.	198	10	160	315	295	41	170	63	55	180	292	—	11.4	6.8	L.A.H.	—	—	—	—	—	—	—
1.30	198	8.75	160	312	290	41	180	63	55	180	292	—	12.6	5.6	L.A.H.	—	—	—	—	—	—	—
2 p.m.	198	8.75	160-1	323	290	40.8	177.4	68.1	44.2	180	288.6	—	11.3	7.0	—	—	—	—	—	—	—	48.1
Aver.																						
2.35	198	12	160	310	263	43	170	62	59	175	280	—	7.2	0.0	L.A.H.	—	—	—	—	—	—	53
3.0	198	12.5	160	307	307	42	160	60	52	170	260	—	11.4	4.8	—	—	—	—	—	—	—	49
3.30	198	11.5	160	313	313	44	195	60	52	180	295	30	10.0	0.0	—	—	—	—	—	—	—	54
4.0	198	12	160	325	303	42	187	75	55	170	275	—	10.6	—	—	—	—	—	—	—	—	51
4.30	198	11.5	161.2	330	300	42.8	186.4	63.4	55.2	175	287	30	10.6	7.5	—	—	—	—	—	—	—	53.4
Aver.																						

Normal load (12,000 lb. per hour from and at 100° C.)

## TEST I.

	In 5½ hrs.	per br.	per lb. coal.
Coal .....	65 cwt.	1844 lb.	—
Water from 44° C. to 160 lb. and 108.6° C. super heat ..	5345 galls.	9718 lb.	7.23 lb.
Water from and at 100° C. ..	—	12800 lb.	9.15 lb.
Boiler efficiency = 76.9%.			

## 30% Overload.

## TEST II.

	In 2 hrs.	lb. per hr.	per lb. coal.
Coal .....	31 cwt.	1736	—
Water from 72.8° C. to 160 lb. and 111.8° C. super heat ..	2712 galls.	13560	7.81
Water from and at 100° C. ..	—	16544	9.55
Boiler efficiency = 80.0%.			

## Distribution of heat.

	Normal.	Overload.
Retained by boiler alone .....	68.3	69.1
Retained by superheater .....	6.8	9.1
Retained by air heater .....	1.8	1.9
Loss in flue gases .....	16.8	17.9
Loss by radiation .....	6.8	2.1

In neither test is the boiler credited with heating the water from the temperature in the water-main (12° C.) to the temperature in the feedwater tank, nor is it debited with the power consumed by the pulveriser (20 H.P.).

Mention has been made previously of a portion of the coal passing through the boiler and making its escape without being completely burned. Doubtless this is due to the larger particles of the coal dust not remaining in the boiler furnace sufficiently long to ensure their complete combustion. Theoretically it is true that the larger particles will be swept out of the combustion chamber sooner than the finer dust. It may be assumed that the coal dust and air issue from the burner nozzle at the same velocity, the coal dust only being about one-twelfth of the weight of the air and many times less by volume. The finer dust will soon be completely burned and lose considerable weight, whereas the heavier particle, in spite of its original momentum, will tend to be stopped more quickly in its ascent by gravity and, turning downwards, accelerate its exit from the combustion zone, even exceeding the velocity of the downward stream of gas. This view is supported by examination of the interior of the boiler after a long run. The fire-brick lining is seen to be most worn away rather more than half way up the tubes, the conclusion being that a considerable proportion of the burning mixture has mushroomed over at that height.

It was thought by reducing the air supply some what that a higher temperature might be maintained in the furnace, and the larger particles more rapidly burnt. Unfortunately the coal in use at the time was very wet, clogging in the pulveriser and on the sieve occurred, and the question has not yet been decided.

This fact emphasises the part played by the hot air in the drying of the fuel as it is crushed.

The boiler has good steaming properties, but in



common with all high temperature boilers, soft water should be used to avoid scaling and consequent overheating of the tubes. Full steam pressure (160 lb.) can be raised within thirty minutes from lighting off, and the manipulation is very simple, the firer only having to alter the mechanical regulation of the coal and water feeds according to the variation in load.

No inconvenience from floating coal dust has been observed, the boiler house being clean and tidy.

Acknowledgment is made to The Directors of the Farnley Iron Co. for permission to include the results of the tests, and to Messrs Fraser and Chalmers, Ltd., for the use of the diagram.

#### ADDENDUM.

The method of obtaining the balance sheet of heat in the various parts of the boiler depends on temperatures and analyses of the flue-gases being taken at the same time at various points. The relationship of these to a conventional temperature of combustion worked out by Bunte, gives the loss of heat in the flue-gas at that particular point as a percentage of the total heat of combustion of the fuel. Correlated with figures obtained from the total heat in the steam raised, all in terms of the percentage of heat in the fuel, a simple calculation then separates the heat retained in the various parts of the boiler.

Thus in the present example on normal load:

	Heat lost from.	Heat retained by.
	%	%
(A) Superheater (calc. from superheated steam tables) .....	—	6.8
(B) Boiler, super-heater, radiation (entering air heater) .....	18.6	81.4
(C) Boiler, super-heater, radiation, air heater (leaving air heater) ..	16.8	83.2
(D) Boiler, super-heater, air heater, (heat in steam) .....	—	76.9

Therefore:—

$$\begin{aligned}\text{Loss by radiation} &= C-D &= 6.3 \\ \text{Retained by air heater} &= C-B &= 1.8 \\ \text{Retained by super-heater} &= A &= 6.8 \\ \text{Retained by boiler} &= B-(6.3+6.8) &= 68.3 \\ \text{Loss in flue gas} &= (100-C) &= 16.8\end{aligned}$$

100.0

#### DISCUSSION.

The CHAIRMAN called attention to the economic importance attaching to the utilisation of small coal, and to the practical difficulties which were encountered in dealing with dust in boilers, gas-producers, and coke-ovens respectively.

Mr. Moss asked what was the horse-power of the pulveriser?

Mr. B. G. McLELLAN said that he had made some tests on a Bettington boiler a few years ago. With good coal it had been frequently observed that the cost of preparing coal, drying, grinding, etc., more than counterbalanced the increase of efficiency obtained. With low-grade coal, which was the rational fuel for this type of firing, there might be a greater margin, but then there was a tendency for the flame to go out, and for the combustion to be incomplete, as was shown by the clinker being accompanied by particles of coke.

For this type of boiler where the flame temperature was intense, the use of a soft water was imperative. In absence of a natural supply, this meant an efficient water softener under regular chemical supervision. As evaporation proceeded, concentration of salts took place in any boiler using softened water. In Lancashire boilers and water-

tube boilers, with tubes arranged to provide circulation of the water, this might be kept under control, but where there was no definite circulation of water, intense local concentration might occur with crystallisation of salts and as a result, burning or explosion of tubes.

The fact that there was not a definite course of circulation, constituted a distinct weakness in this boiler.

It would be interesting to know the author's experience on the life of the firebrick lining, also if any attempt had been made to employ a lining of the same type as the ash of the coal in use, i.e., an acidic brick for coal with an acidic ash, and *vice versa*.

Mr. WILLIAM MCD. MACKEY asked if the boiler was intended for a poor coal, i.e., a coal high in ash, or was it intended that good coal should be ground. In the case of a poor coal he considered that it was better to wash it and then use it in the ordinary way as a boiler fuel, or grind and coke it. In pit practice about half the coal was sold at above cost price, and the rest, the small coal, below, and it was this that was wanted for coke making and ordinary boiler fuel; but the smallest, the fine slack and smudge, contained the bulk of the dirt or ash. The dirt came from the partings in the seam, from the roof, and from the floor, and could be readily washed out of the small coal, except in the rare cases in which the dirt was intergrown with the coal. He agreed with Mr. McLellan's remarks with reference to the action of the fine ash on the brickwork of the furnace.

Mr. C. P. FINN asked for a description of the disintegrator and also what amount of moisture caused this to clog. In coal washeries the fine dust washed away from the coal was allowed to settle out from the water; he wondered whether this would be of any use with these boilers.

Mr. F. MILLER said he understood that it was impossible to store coal dust owing to risk of spontaneous heating. In the present instance, however, the coal was ground immediately before use. He also had seen it stated that it was impossible to screen the coal if it contained more than 1% of moisture. What was the life of the firebrick lining?

Mr. DOUGILL asked if the boiler was adapted to fine coke breeze?

The AUTHOR replied that it was intended to show an improved method of steam-raising by using low-grade small fuel, though with good coal an increased efficiency over ordinary practice would be obtained. Fine coal, which was otherwise wasted, either in the mine itself or on washing plants, could be used if sufficiently dry. Coal containing up to 14% of moisture had been successfully burned though necessitating a rather large excess of air to prevent clogging in the pulveriser. It was pointed out that air at 60° C could carry 19% of its volume of water vapour, much more than was contained in the coal, and the dust passing to the screen was found by direct determination to be perfectly dry. Coke breeze would have to be much more finely pulverised than coal to be satisfactorily used.

The pulveriser was of the "rotary" type, the blades lasting about three months. During the first test the power consumed by it was 28 EHP, which would correspond with approximately 3% of the steam raised, assuming 15 lb. of steam per kilowatt. No tendency for the flame to go out had been noticed during a period of two years' working.

Definite circulation of water in this boiler was doubtful, and the necessity for the use of soft water had been noted. The life of the brick lining was not a simple question of the acidity or basicity of the ash as compared with the firebrick, though that was important, the physical structure and previous treatment of the clay being also a prime factor.

## Communications.

### NOTE ON GHEE.

BY H. C. BROWNING, M.A., AND M. PARTHASARATHY  
(Government Analyst's Laboratory, Colombo,  
Ceylon).

In two papers (Analyst, 1910, p. 343, and 1911, p. 392) Bolton and Revis give figures for the Reichert-Meissl value of ghee. They state that it is usually approaching or even exceeding 30.

Kesava Menon (J. Soc. Chem. Ind., 1910, 1428) finds a Reichert-Meissl value of 25.7 for cow ghee and 18.24 for buffalo ghee. These samples were made at the author's home and were thus genuine.

K. H. Vakil (J. Soc. Chem. Ind., 1915, 320) finds that the Reichert-Meissl values vary from 26 to 25.

The writers of this note have frequently had samples of ghee which appeared to be genuine (from the analytical figures) but which had Reichert-Meissl numbers lower than those of Bolton and Revis.

They have therefore prepared samples of ghee in the laboratory, from cows and buffaloes milked under their personal supervision.

The following is a typical example:—

Sample.	Origin.	Butyro-refractometer reading at 40° C.	Oleo-refractometer at 45° C.	m.p.	Acid value.	Reichert-Meissl value.
1	Buffalo	44°	—25°	43°	0.83	18.9
2	Cow	45.4°	—23°	38°	0.48	22.3

The Reichert-Meissl numbers of four buffalo ghees prepared in the laboratory were 18.9, 18.86, 27.0, and 30.2.

Reichert-Meissl numbers of cow ghees also prepared in the laboratory were 21.4, 22.3, 23.9, 20.9, and 20.9.

Ghees prepared from imported frozen Australian butter had Reichert-Meissl values 29.4 and 28.0 respectively.

It will thus be seen that it is quite possible for genuine ghees to have Reichert-Meissl values well below 30.

The writers' experience is that the oleo-refractometer is most valuable in the preliminary examinations of ghees.

### ORGANIC VULCANISING ACCELERATORS.

BY D. SPENCE.

In view of the world-wide publicity given in recent times to the above subject it would seem to be no longer amiss, but, indeed, desirable to put on record a few facts in refutation of some of the very erroneous statements which have appeared in the literature of this subject. The various writers reviewing this subject, and in particular Dittmar,<sup>1</sup> Gottlob,<sup>2</sup> King,<sup>3</sup> Peachey,<sup>4</sup> have all, without exception, assumed that that which is not proclaimed from the house top is not known, and from these faulty premises have drawn certain equally erroneous yet sweeping conclusions which it has been my intention sooner or later to nullify and correct. I shall take the various references in the order in which they appear.

Dittmar<sup>1</sup> in that part of his article which deals with the subject of organic accelerators refers to the first patent application<sup>2</sup> on this subject as follows:—  
“The 16th of November, 1912, marks an important turning-point in the development of the rubber industry. For on the publication of this patent there began a systematic hunt in German, Russian,

Austrian, and in American rubber factories for organic vulcanising accelerators. The ice was broken; the practical value was recognised.” This patent deals with the vulcanisation of natural or of synthetic rubber using piperidine or its homologues as accelerators.

In similar, if somewhat more modest, language Gottlob<sup>2</sup> describes the discoveries of the Elberfeld Farbenfabrik, and in all the subsequent patents dealing with the subject of the vulcanisation of natural or of synthetic rubber by this company the novelty as well as the scope of their discoveries are described in no uncertain terms.

In the article by King<sup>3</sup> dealing with this subject it is stated that the use of organic accelerators dates from the discovery of synthetic rubber. “This product cannot be vulcanised without the addition of some of the organic catalysts.” . . . “The first patent granted for an organic accelerator was to Bayer & Co. (German Patent 265,221, November 16th, 1912) for the use of piperidine.”

According to all these authorities then the discovery of the principle of vulcanisation whether of natural or of synthetic rubber by means of organic catalysts dates from the disclosures of the Elberfeld Farbenfabrik in November, 1912. And by reason of the sources of the information I am inclined to believe that this is true so far as Europe in general and Germany in particular are concerned. But as one who has studied this aspect of vulcanisation, continuously, for the past several years, both scientifically and industrially, I must take exception to their generalisations as far as America is concerned. The “systematic hunt” which Dr. Dittmar refers to, took place in America not after but several years previous to the first disclosure in German patent literature to which he refers, and I hope to demonstrate that this field had been covered and the scope of the discovery determined long before the Elberfeld Farbenfabrik had even commenced to investigate this subject. All their claims as well as those of others to novelty in this connection can readily be disposed of, coming as they do at this late date.

Substantial proof of my assertion is indeed to be had from a careful analysis of my own previously published work on the subject of vulcanisation. In the *Kolloid Zeitschrift*<sup>5</sup> of 1912, more than six months before the Elberfeld Farbenfabrik made application for their first patent on this subject I described certain experiments the significance of which passed apparently unobserved even by the chemists of the Elberfeld Farbenfabrik. In this communication I wrote as follows:—“More than a year ago we prepared samples of two carefully-chosen mixings. Both mixings were made from partially purified rubber with about 9% of sulphur. In order to make the investigation as comprehensive and complete as possible the two mixings were purposely chosen so that two quite distinct types of compound as different as possible in their properties were obtained. The first was a slow-curing compound deteriorating rapidly; the second, on the other hand, was a quick-curing mixing which we knew from experience would resist deterioration indefinitely. . . . Two compounds of the same general character as No. 2 were prepared; both these compounds contained about 9% of sulphur and were free from mineral compounds.”

A moment's consideration of the statements italicised by any chemist of india-rubber familiar with his subject should at once make evident the fact that the acceleration of vulcanisation in the two compounds described must have been produced by novel methods, by substances organic in nature, for the use of litharge, magnesia, or of any of the accelerators then known is expressly precluded. As a matter of fact the first of these two compounds actually contained 1% of piperidine, the very substance referred to in the German patent of November 16th, 1912; the

formula for the mixing was as follows:—Plantain Para 100 pts., sulphur 10 pts., piperidine 1 pt. (about the use of piperidine in this mixing the results given in Table V. of the communication referred to would have been utterly impossible, as I saw full well before I made the test, while debating in my mind as to the best type of compound and accelerator to use for these experiments on vulcanisation at low temperatures. Now in regard to the discovery of vulcanisation of synthetic rubber by means of organic accelerators, also accredited to the Elberfeld Farbenfabrik. The vulcanisation of these products by this means was actually carried out and successfully accomplished by me both for isoprene-caoutchouc and for the dimethylcaoutchouc from pinacone at a time when the large industrial concerns in Germany engaged in the study of the synthesis of indiarubber were in the dark as to their lack of success in the vulcanisation of their products. The proof of this assertion and the complete vindication of my position with reference to the subject in general I am content to leave over until a later date. At that time I will also bring evidence to show that the reagents claimed by Peachey (English Patent 4263 of 1914) were known to me and had been used by me both scientifically and industrially several years before his application was applied for. In conclusion, although it cannot be denied that Germany has taken the lead in many of the discoveries of science in its application to industry and the present instance has again sought to establish her priority in invention. I believe that I have already brought out enough evidence to refute her claims in this instance and to show that, notwithstanding her silence, America has not been asleep on the contrary took the lead in the discovery of this one fundamental principle of vulcanisation which has only now become universally recognised.

- <sup>1</sup>Hitmar, Gummi-Zeitung, 29, [17], p. 425 (1915).
- <sup>2</sup>Kolloid, Gummi-Zeitung, 29, [14], p. 303 (1916).
- <sup>3</sup>King, India Rubber Journal, 52, [13], p. 440 (1916).
- <sup>4</sup>Peachey, India Rubber Journal, 52, [18], p. 603 (1916).
- <sup>5</sup>Ger. Pat. 265,221, Nov. 16, 1912, Elberfeld Farbenfabrik.
- <sup>6</sup>Kolloid Zeitschrift, 10, 303—305 (1912).

## Industrial Notes.

### COMMITTEE ON GLASS AND OPTICAL INSTRUMENTS.

The Advisory Council of the Government's Department of Scientific and Industrial Research has added to the list of their technical Committees a Standing Committee on Glass and Optical Instruments. The membership of the Committee is as present as follows:—Professor H. Jackson (chairman), Mr. Conrad Beck, Professor C. V. Raman, F.R.S., Mr. F. J. Cheshire, Mr. A. E. Conrady, Mr. A. S. Esslemont, Mr. J. W. French, Dr. R. T. S. Zbrook, F.R.S., Sir Howard Grubb, F.R.S., Mr. E. B. Knobel, Dr. T. R. Merton, Professor W. Nicholson, Captain Creagh Osborne, R.N., Mr. H. J. Stobart, Mr. J. Stuart, Mr. M. P. Swift, Mr. William Taylor, Mr. F. Twyman, Lt.-Col. C. Williams, Mr. W. F. J. Wood. The Committee met on December 11th, and, in regard to the urgency of the problems requiring investigation in respect of these essential industries, appointed a series of Sub-Committees, of which various special problems were referred. Among these problems the more important are:—  
(A) Raw materials for glass and glass making.  
(B) Optical properties of a large range of glasses.  
(C) General physical and chemical properties of glass and glassware for scientific and industrial purposes.

- (D) Testing and standardising of glassware.
- (E) Workshop technique.
- (F) X-ray glass apparatus.
- (G) Optical calculations and lens designing.
- (H) Optical instruments.
- (J) Translation of foreign works on optics.

This brief description indicates certain lines of investigation which have been brought forward. The Standing Committee does not propose to limit itself to these subjects, but is prepared to consider and report upon the necessity for investigation in other directions, relevant to its terms of reference. Manufacturers who have experienced difficulties requiring investigations for their solution in connection with the subjects of glass and optical instruments or who desire to make suggestions for special researches on these subjects are invited to communicate in the first instance with the Secretary of the Research Department, Great George Street, Westminster, S.W., who will direct the correspondence into the appropriate channels for attention.

### ELECTROMETALLURGY IN THE UNITED STATES IN 1916.

According to an article in the Jan. 1st issue of *Metallurgical and Chemical Engineering*, there was a greater commercial development of electrometallurgical industries in the United States during 1916 than at any time during recent years. The heavy consumption of alloy steels formerly made largely in the crucible, the increased demand for ferro-alloys caused by the greater production of alloy steels, the shutting-off of imports of ferro-alloys and other electrometallurgical products, like aluminium and magnesium, the high prices of metals permitting experimentation with new processes—all these are contributory causes of the recent industrial advance in electrometallurgy.

The installations of electric steel furnaces in America were approximately doubled in 1915, and the progress continued at almost undiminished rate in 1916. The chief trouble met with is a lack of experienced workers. High-speed tool steel is now being manufactured in 8-ton heats as a regular procedure, in contrast with the 100-lb. pour of the crucible. The heavy demand for high-speed tool steel and the difficulty of securing crucibles at any price has made the electric steel furnace an economic necessity.

A considerable number of electric furnaces have been installed in foundries for the manufacture of steel castings. More installations are contemplated involving the use of the electric furnace in a duplex process with the open-hearth or the converter. Several electric furnaces are melting ferromanganese before addition to steel.

In Germany—formerly the largest producers of electric-furnace steel—the heavy demand for tool steel has also resulted in an increased use of the electric furnace for steel making. In August the production reached 17,000 tons per month, or six times the monthly production at the outbreak of the war. On the other hand, the production of crucible steel in Germany has remained stationary, there being no new installations of any size. The monthly production of electric-furnace steel in Germany is now double the crucible output.

On a smaller scale there has been a heavy increase of ferro-alloy manufacture in the United States. Three new manufacturers of ferrosilicon started production during the year. At the outbreak of the war about half of the domestic ferrosilicon consumption was imported, but now practically all ferrosilicon needed for consumption is manufactured in the country, and a small amount is being exported. To provide part of the needed furnace capacity a calcium carbide plant in

Niagara was converted to ferrosilicon manufacture. During the period in question the domestic consumption of ferrosilicon has doubled and is now estimated at 45,000 tons annually.

The largest domestic producer of ferrochrome was able to supply the increased needs of the steel industry, but it is probable that if this producer had not held large stocks of foreign ore, there would have been a shortage of ferrochrome. The development of California mines has been slow and the average grade of the chromite was too low for alloy manufacture. With other alloys, such as ferrotungsten, ferrovanadium, ferrotitanium, and ferromolybdenum, there has been an increase of production and several plants have been established. There is now sufficient plant capacity in the country for filling all domestic requirements of ferrotungsten, and for the first time considerable quantities of the alloy are being exported. Exports of ferrovanadium have doubled since 1915. Most of the new producers are using the electric furnace. Ferrotitanium is produced at Niagara to the limits of plant capacity. A fair quantity of ferromolybdenum is being manufactured in the United States, and towards the end of the year there was a marked increase in demand for foreign shipment, which is believed to have to do with the lining of big guns. Ferro-uranium was produced commercially for the first time in the States, and may be added to the new products of the electric furnace.

Aluminium manufacture in the United States enjoyed in the past year maximum production and maximum prosperity in its history, the lead over European producers being increased. The North Carolina plant of the Southern Aluminium Company, which in 1915 passed into the hands of the Aluminium Company of America, is expected to start operation in the near future. Further large projects for increased plant capacity are under way. The imports dropped to about one-tenth of the normal quantity and exports doubled. There are no new producers besides the Aluminium Company of America, up to the present. J. W. Richards estimates that by 1925 aluminium will be third in importance among metals (measured by weight), and will be outranked only by copper and iron.

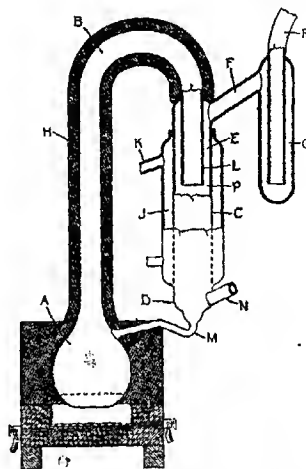
As a result of operations started in 1915 several plants are producing very pure metallic magnesium on a small but commercial scale by electrolysis.

Among new electrometallurgical enterprises electrolytic zinc looms large. There is the plant of the Anaconda Copper Company at Great Falls, Montana, with a production of 100 tons of electrolytic zinc per day, while as a result of the Bully Hill experiments of the General Electric Company a \$350,000 electrolytic zinc plant is being erected at the Mammoth smelter in California. As in 1915, there was in 1916 practically no development of electric-furnace smelting of zinc ore. It would seem that electrolytic zinc has the upper hand, and may be able to keep it, although in a measure its sudden development to a commercial process has probably been accelerated by the facilities of the Anaconda company available for placing an experimental development on a commercial basis.

The electrolytic copper refineries that were hit so hard at the start of the war have all been working to the limit of their capacity. Electrolytic refining has also been successful with tin; it has often been stated that Bolivian tin ores would always be out of question as the tin produced would be too impure; the success of the combined tin smelting and electrolytic refining plant of the American Smelting and Refining Company at Perth Amboy has proved that the additional refining cost is by no means prohibitive, and the only reason why enlargements have not yet been made is the present high cost of building materials.

## HIGH VACUUM PUMPS.

In a recent issue of the *Journal of the Franklin Institute*, Dr. Irving Langmuir, of the General Electric Company, Schenectady, describes a new form of vacuum pump, which is character-



Glass condensation pump.

by extreme speed and the high degree of vacuum attainable. In these pumps the gas is carried into a condenser by means of a blast of mercury vapour, the action being similar to that in a steam ejector and in a Gaede diffusion pump. The method by which the gas is brought into the mercury vapour blast in the new pump is based on a principle which differs essentially from the employed in the steam ejector or Gaede pump. In the new pumps, which are called "condensation pumps," the gas to be exhausted is caught in the blast of vapour and is forced by gas friction to travel along a cooled surface. By maintaining this surface at such a low temperature that the condensed mercury does not re-evaporate at a appreciable rate, it is possible to keep the mercury vapour from escaping into the vessel which is being exhausted. The action of the pump therefore depends primarily on the fact that all the atoms of mercury striking a mercury-covered surface are condensed, no reflection from the surface taking place. The pumps can be worked at very high speed (3000-4000 c.c. per second, or even more), and there is no lower limit, other than zero, to which the pressure may be reduced. By means of this pump, pressures lower than  $10^{-4}$  mm. have been produced and measured.

One form of the condensation pump, made entirely of glass, is illustrated here. Mercury vapour from the flask, A, is carried through the thermally insulated tube, B, to the nozzle, L. The vessel to be exhausted is connected to R. The gas from this vessel passes through the trap, C, and tube, F, into the annular space, E. As this gas comes into contact with the mercury vapour blast issuing from L, and is thus forced outwards and downwards against the walls of the tube, C, and is finally driven down into the space D, from which it escapes into the rough pump connection, N. The mercury which condenses on the sides of the tube, C, passes back through M into the boiler, A. This construction prevents the condensed mercury passing into the annular space, E, and thus the trouble arising from a mercury blast into the tube, F, is avoided. It

essential that the end of the nozzle, L, should be below the level of the water in the condenser, J. The distance between L and D must be sufficiently great that no perceptible quantity of gas can diffuse back against the blast of mercury vapour, and so that a sufficiently large condensing area is furnished. The other dimensions of the pump are relatively unimportant. The pumps may be made of any size. The larger the pump the greater will be the speed of exhaustion. Such pumps have been constructed with the tube, B, and nozzle, L, in sizes varying from  $\frac{1}{4}$  inch to  $1\frac{1}{2}$  inch.

In working the pump, the mercury boiler, A, is heated either by gas or electrically so that the mercury evaporates at a moderate rate. A thermometer in contact with tube, B, under the heat insulation, usually indicates a temperature between  $100^{\circ}$  and  $120^{\circ}$  C. when the pump is working satisfactorily. Under these conditions the mercury evaporates quietly, and no bubbles are formed, so that there is no tendency to pumping. With an electrically heated pump in which the nozzle, L, was  $\frac{1}{4}$  inch diameter, the pump began to work satisfactorily when the heating unit delivered 220 watts, but the speed of exhaustion remained practically unchanged even when the heating current was increased to about 550 watts.

The back pressure against which the pump will operate depends, however, on the amount and velocity of the mercury vapour escaping from the nozzle. Thus with 220 watts the pump mentioned would not operate with a back pressure exceeding 60 bars (bar = 0.0075 mm. mercury pressure), whereas with 500 watts back pressures as high as 800 bars did not affect the working.

The condensation pump, like any other mercury pump, does not remove mercury vapour from the system to be exhausted. (The vapour pressure of mercury at room temperature is about 2 bars.) By the use of a trap such as that shown at G, this vapour pressure may be lowered, and mercury vapour can be entirely eliminated by the use of solid carbon dioxide or liquid air.

The condensation pump also lends itself to construction in metal, and two types of metal pumps are illustrated in the article referred to (J. Franklin Inst., 1916, 182, 719-743).

#### PLANT DEPRECIATION.

In a paper before the Institution of Electrical Engineers, Messrs. F. Gill and W. W. Cook discussed the principles underlying the computation of depreciation of plant. Neither the engineer, nor the financier, nor the accountant is competent to settle the question in any particular case, because to obtain correct results there must be co-operation among the three. The function of the engineer is to provide the information necessary regarding the plant, such as first cost, residual value, and life. The financier has to furnish information regarding the money, the amount available, the rate at which it can be borrowed, and the rate that can be obtained for the loan of it. The accountant has to provide accurate records based on the decisions arrived at, and to see that the real results are neither disguised nor hidden.

Considering that this common method can best be studied in the annual charges for plant, determined in advance of construction, these are analysed into four principal parts:—(1) Return on capital outlay, treated as a charge, because no engineer is justified, except in special circumstances, in putting down plant that will not pay a return on its own capital; (2) depreciation, including "renewals" or provision for diminution in value of plant in place and working by reason of causes outside the owner's control, such as age,

wear, and accidents, and "improvements" to enable the owner to take plant out of commission before its physical life is exhausted in cases where, either from progress of the art or growth of the business, it is economically advisable to do so; (3) maintenance, or the cost of keeping plant in as good condition as is advisable, but not of renewing it; and (4) operation, the cost of working the plant. To determine the amount in the annual charges for depreciation it is thus necessary to estimate the first cost, residual values, and the physical and economic lives of the different classes of plant. The treatment of the capital account must also be fixed and the charge for return on capital estimated.

The reserves are preferably handled by means of two funds, for "renewals" and for "improvements." The calculation of the amounts to be contributed to these two funds must include the interest which should be earned by the money before it is required at the end of the life. The amount which should be in the renewals fund gives the correct diminution in value, when considering the value to the owner, of the plant in place and working, at any period in its history. The amounts annually put to the two funds, renewals and improvements, determine the correct charge against the undertaking for the purpose of assessing profits, fixing rates, and, under certain conditions, assessing the value of the undertaking as a going concern.

There are several methods of treating the provision for depreciation, but substantially they reduce to the following:—(1) Maintenance method, in which no regular sum is put aside, the plant being maintained and renewed out of revenue expenditure; (2) reducing balance method, in which a uniform percentage is deducted each year off the reduced capital cost; (3) straight line method, in which the annual charge is the ratio of one year to the total time or life; and (4) sinking fund and annuity method, in which value is given to the interest the fund will earn until it is wanted.

The first method affords no means of calculation, is bound to be irregular, and since no charge can be made until a renewal actually takes place, is always in arrear unless and until a complete renewal is effected. The second is very frequently employed to ascertain the amount to be allocated for depreciation at the end of a year, but it results in a relatively heavy charge in the early years and a very light one in the last years.

The third method, as generally used, is very simple. Assuming the first cost to be £100, the residual value £15, and the economic life 19 years, then £87 has to be found in that period, and therefore all that is required is £4.58 per annum for depreciation, the total charge, with £5 for return, being £9.58 per annum. But the present value of 19 annual payments of £9.58 at 5% is £115.77, instead of £94.86, and the method does not pass the test of total cost. If £4.58 be set aside annually, £87 will be accumulated at the end of 19 years, but since it is not prudent to leave money idle, interest ought to be and will be earned by the money set aside. If interest at 5% is added to the growing fund, there will be at the end of the 19 years not the £87 required, but £138.84; hence the yearly payments must be reduced below £4.58 until the amount is just sufficient to accumulate only the £87 required. This needs an annual payment of £2.85 for the 19 years.

If the earned interest be not added to the fund, but credited to the general revenue account, as is generally the practice of those who uphold the straight-line method, the effect is to increase the revenue, with the result that a higher return on capital is obtained, the interest earned by the depreciation fund not being required for the plant. This is a violation of the hypothesis that a stated return on capital is to be earned, no more and no less, and the present value of all the

payments exacted for the plant is too great, being \$115.77.

If instead of accumulating a fund the capital is partly repaid each year, the present value is correct at \$94.86, but the annual charges vary each year, and there is no condition as regards the plant which requires a varying annual charge.

In the annuity method a part of the capital is repaid each year and a return is paid on the capital outstanding, while in the sinking fund method the capital remains fixed at \$100 and a fund is built up gradually against the end of life, at which time, and not till then, the money will be required either to repay the capital or to replace the plant. In both methods the total charge is uniform at \$7.85 per annum, made up in the former case of the decreasing return component and an increasing depreciation component, and in the latter of uniform charges each year both for return and for depreciation. In both methods the present value of the series of components is \$94.86. Thus they are both correct in amount, satisfy all real requirements demanded by the plant, and being uniform can be readily handled. In practice it is not usual to repay the capital fractionally each year; the money set aside for depreciation is generally accumulated until it is wanted, and the sinking fund method satisfies all requirements so far.

### ELECTROLYTIC HYDROGEN AND OXYGEN.

In a recent issue of the *Engineering and Mining Journal* L. V. Curran gives an account of the operation of an electrolytic hydrogen and oxygen plant at the Copperhill property of the Tennessee Copper Co.

The principal equipment is as follows: One 25-kw. 80-volt direct current generator, direct-connected to one 30-h.p. 220-volt 60-cycle three-phase 900 r.p.m. slip-ring-motor; fifteen oxygen-hydrogen generating cells; two 50-cu. ft. capacity gasometers; two  $6\frac{1}{2} \times 3 \times 5$ -in. two-stage tandem belt-driven compressors, 200 r.p.m.; one 15-h.p. 220-volt 60-cycle three-phase 1150 r.p.m. induction motor; ten  $15 \times 48$ -in. drums, tested to 1000 lb. water pressure, fitted with right-hand connections; fifty-four  $15 \times 48$ -in. drums, tested to 1000 lb. water pressure, fitted with left-hand connections; two  $4 \times 10$ -ft. receivers tested to 300 lb. water

pressure. The plan of the plant is shown in the accompanying figure.

The cells consist of an iron tank  $40 \times 6 \times 47$  in. high, forming the positive electrode, and two  $\frac{1}{8}$  in. iron plates,  $36 \times 39\frac{1}{2}$  in. high, forming the negative electrode, an asbestos fabric entirely inclosing the latter and preventing the gases from mixing. The cells are spaced on 9-in. centres and enclosed in a cellroom  $18 \times 9 \times 8\frac{1}{2}$  ft. high. The water is kept charged with sufficient caustic soda to maintain a density of  $26^\circ \text{B.}$ , about 75 lb. per cell being required at first filling.

The most economical rate of generation with these cells is reached when the current is 400 amp.; so the current is kept at that strength, 40 volts being required when cold and 36 volts at the running temperature, which is about  $127^\circ \text{F}$  ( $54^\circ \text{C.}$ ). In order to minimise the danger of the gases getting mixed, the equipment for collecting the gases from the cell tops and for compressing into the drums and receivers is arranged entirely in duplicate, with no cross-connections, the scheme of left-hand and right-hand connections being carried out on the compressor discharges and drum connections, for hydrogen and oxygen respectively. Further precautions are taken by watching the polarity of the generator and by making frequent tests of the oxygen.

The gases are collected into  $1\frac{1}{2}$ -in. manifolds with rubber-hose connections to each cell, and are led into two gasometers which, as often as filled, are pumped down by the two compressors driven by the 15-h.p. motor through a lineshaft. The oxygen is pumped into drums and used for welding and cutting and for certain heavy lead work.

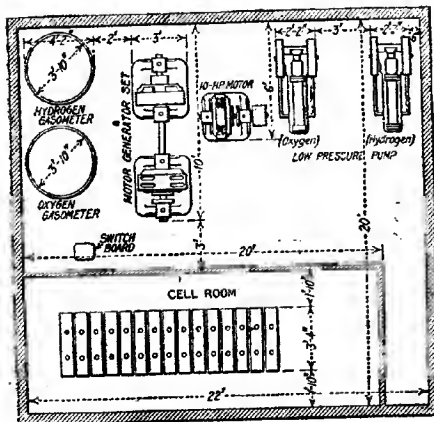
The hydrogen is pumped into the "hydrogen system," consisting of two  $4 \times 10$ -ft. receivers, one at the gas plant and one at No. 1 sulphuric acid plant, approximately 800 ft. distant, the two receivers being connected by a  $\frac{1}{2}$ -in. line. From the acid-plant receiver the gas is piped through the plant in  $\frac{1}{2}$ -in. and  $\frac{1}{4}$ -in. lines, being used at 4 lb. pressure. The pressure carried on the gas-plant receiver is 200 lb. and on the acid-plant receiver, 50 lb. Hydrogen is also compressed in drums for use on work at the other plants or in places isolated from the hydrogen system. Compressed air is used with the hydrogen for lead work except where there are no air lines or where the work is particularly heavy, when the oxygen is used. Hydrogen is also used with oxygen at the welding shop in cutting.

The output of the plant was found to be  $12\frac{1}{2}$  lb. of hydrogen and  $97\frac{1}{2}$  lb. of oxygen per 24 hr. The present hydrogen consumption is  $14\frac{1}{2}$  lb.,  $5\frac{1}{2}$  days per week. It is therefore necessary to operate the plant  $153\frac{1}{2}$  hr. per week, or  $91.4\%$  of the time. The present demand for oxygen is  $19\frac{1}{2}$  lb. per day, six days per week. Current is bought at \$30 per h.p.-yr.; the power input of the plant is 26.25 h.p.

The first cost of the plant was \$6287.42. The present operating costs are:—

Yearly operating costs.	
Power, $26.25 \times \$30 \times 91.4\%$ .....	\$719.78
Engineers, 8007 hr. at 18¢c. ....	1437.66
Interest and depreciation, 12% .....	754.46
Inspector lines, 4556 hr. at 16¢c. ....	728.96
Total yearly expense .....	\$3721.46

At the present rate 6022.5 lb. of oxygen and 4086.9 lb. of hydrogen are used per year. The cost of replacing the service with bought oxygen and hydrogen generated with zinc and acid would be about \$17,000.





## Journal and Patent Literature.

PATENT SPECIFICATIONS may be obtained by post by remitting as follows:—

English.—*sd.* each, to the Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C.  
 United States.—*ls.* each, to the Secretary of the Society, who has to furnish the U.S. Patent Office with the following data:—  
 Patent number, date, name of patentee, and title of invention.  
 French.—1 fr. 05 c. each, as follows: Patents dated 1902 to 1907 inclusive, Belin et Cie., 56, Rue Feron 8, Paris (3e.)  
 Patents from 1908 to date, L'Imprimerie Nationale, 87, Rue Vieille du Temple, Paris.

### I.—GENERAL; PLANT; MACHINERY.

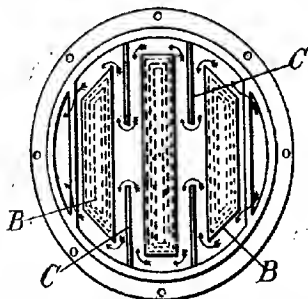
#### PATENTS.

*Iron and similar bodies; Device for freeing from impurities.* C. Hülsmeier, Düsseldorf-Grafenberg, Germany. Eng. Pat. 8134, Mar. 31, 1914.

THE steam is passed in an upward direction through several separate spiral passages contained in a conical casing. The latter is enclosed in an outer casing and is provided at the top with an outlet for the purified steam. The inlets to the spirals are closed by ball valves of different heights, the lightest of which opens when only a little steam is passing, the next heavier when more steam is passing, and so on. The edges of the spirals have bent-over channels to facilitate the discharge of the oil or other impurities. The latter pass through openings in the conical casing into a common pipe for each vertical series of openings, and pass thence into the lower part of the space between the conical casing and the outer casing. An electric lamp is fitted in the purified steam outlet, which is also provided with a window so that the extent to which the steam is purified can be observed.—W. H. C.

*Dephlegmator.* J. E. Mitchell and H. Morley, Barnsley. Eng. Pat. 17,567, Dec. 16, 1915.

A DEPHLEGMATOR tray or section for connection with benzol and other stills is shown in plan in the figure. It is constructed with a number of



apour outlets surmounted by caps or hoods, B, between which baffle plates, C, are interposed to cause the condensed liquid to travel in a circuitous route.—J. E. C.

*Refrigerating apparatus; Absorption.* F. T. Dunsford, and The Seay Syndicate Ltd., Manchester. Eng. Pat. 17,672, Dec. 17, 1915.

A absorption refrigerating apparatus of the type in which ammonium nitrate is employed as an absorbent for ammonia. The absorbent is supported upon a corrugated or ribbed surface which contains the pipes or passages for the heating or cooling media; several such surfaces may be arranged in a tier.—W. F. F.

*Grinding machinery.* T. Breakell, Wirksworth, Derby. Eng. Pat. 102,631, Jan. 11, 1916 (Appl. No. 478 of 1916) and Apr. 6, 1916 (Appl. No. 5089 of 1916).

IN a mushroom type disc mill, a grinding disc is positively driven by a shaft having a universal joint, the disc co-operating with a controlling ring whereby a gyratory motion of the disc is produced. The grinding may be effected partly on the face of the disc and partly on a step or similar arrangement for giving an action corresponding to crushing on the edge of a roll.—J. E. C.

*Crushing machinery.* R. E. Ellis, London. From R. W. Pringle, Gatooma, South Rhodesia. Eng. Pat. 102,767, Feb. 17 and Aug. 11, 1916. (Appl. Nos. 2406 and 11,385 of 1910.)

IN a crushing machine of the type in which the crushing members comprise two concentric rotating mullers of inverted conical form, with the annular space between them converging towards the bottom, the lower ends of the mullers are cylindrical and the outer muller has a flared extension at the bottom. A second inner conical muller is parallel to the flared end of the outer muller and is adjustable axially so as to vary the discharge space between the mullers. The first inner muller may be raised and dropped periodically by a cam operated by the driving mechanism.—W. F. F.

*Kiln; Recuperative.* R. D. Pike, Richmond, Cal. U.S. Pat. 1,207,209, Dec. 5, 1916. Date of appl., June 1, 1915.

THE exhaust gases from the heating chamber pass through a number of openings in the hearth which register with rows of vertical flues through which the gases pass to a main waste gas flue below. The air to support combustion passes through rows of horizontal flues which alternate with the before-mentioned vertical flues, and heat exchange takes place between the gas and air. The air passes backwards and forwards through groups of horizontal flues and enters an annular flue in the wall of the heating chamber, from which it passes to the burners, which are spaced around the periphery of the heating chamber.—W. H. C.

*Centrifugal machine and method.* I. P. Sharples. West Chester, Pa. U.S. Pat. 1,207,230, Dec. 5, 1916. Date of appl., Apr. 27, 1915.

A SUSPENDED revoluble bowl is provided with a bottom inlet pipe, upper outlet pipes, and with a steadying device at the bottom. The lower inlet pipe is connected with a tank containing the liquid to be treated, which is drawn into the bowl by the suction produced by the discharge of the separated liquid through the outlet pipe.—W. H. C.

*[Graphite] lubricants; Method of making and products thereof.* C. H. Bierbaum, Buffalo, N.Y. U.S. Pat. 1,207,843, Dec. 12, 1916. Date of appl., Apr. 7, 1915.

A WET mass of pulverised graphite freed from impurities is mixed with "petroleum," agitated, and diluted with oil and a solution of "petroleum."—W. F. F.



**Graphite crucibles; Composition for preserving**

— J. B. Carmichael, Assignor to H. J. Harper, Seattle, Wash. U.S. Pat. 1,207,858, Dec. 12, 1916. Date of appl., Jan. 28, 1916.

THE composition consists of a mixture of 18 oz. of pulverised glass, 9 oz. of pulverised pumice, and one quart of a saturated solution of sodium silicate.—W. H. C.

**Measuring the flow of fluids; Method and apparatus for**

— C. W. Humphrey, Chicago, Ill. U.S. Pat. 1,208,159, Dec. 12, 1916. Date of appl., Dec. 17, 1915.

THE flow is measured by observing the differences which occur in the ratio between the expansion of a measured quantity of gas enclosed in a chamber under atmospheric pressure and wholly surrounded by the liquid, the flow of which is to be measured, and the quantity of electric current passed through a resistance heater in the enclosed chamber, when the liquid is at rest and when it is flowing at various speeds.—W. H. C.

**Mixing and distributing liquids; Apparatus for**

— O. Schutz, Assignor to Badische Co., New York. U.S. Pat. 1,208,670, Dec. 12, 1916. Date of appl., Feb. 9, 1916.

A TANK having a front wall lower than the other three walls is divided into compartments by cross walls of less height than the front wall and provided at alternate ends with openings. The liquid is fed into the compartment farthest from the front wall, circulates through the compartments, and is discharged by means of capillary material, which is suspended from a bar above the tank and after dipping into the liquid hangs over the front wall of the tank.—W. H. C.

**Dryer; Mechanical**

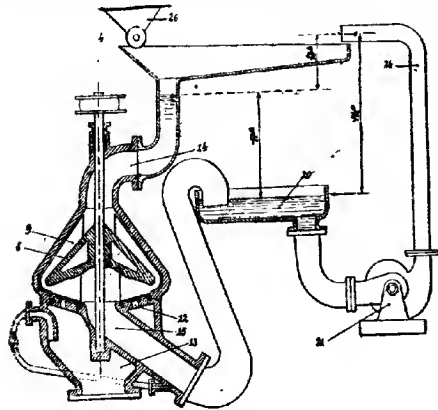
— H. L. Julien. Fr. Pat. 480,100, Oct. 29, 1915.

THE material to be dried is moved by a conveyor substantially horizontally through the dryer. Air is drawn in by a fan at the top of the casing, near the outlet for the material, and passes transversely through the material to a heater at the bottom. The air is passed repeatedly through the dryer from top to bottom, being further heated at each stage, till it finally escapes near the inlet for the material.—W. F. F.

**Separating uniform or mixed materials according to density or size of particles; Apparatus for**

— J. Bernard and C. Vibert. Fr. Pat. 480,376, Dec. 2, 1915.

THE material to be separated is fed from the hopper, 26, to the conduit, 14, supplied with water



by the pump, 21, and pipe, 24. The rotating shaft, 4, carries a member, 8, having blades, 9. By suitable adjustment of the relative actions of centrifugal force and of the current of water, material of any desired density or size of particle may be caused to escape by the holes, 12, to the collector, 13. The water passes on to the conduit, 15, and thence to the tank, 20, which supplies the pump. Several similar separating apparatus may be connected in series.—W. F. F.

**Pulverisation of solid substances; Process for the**

— L. Boulade. Fr. Pat. 480,410, Dec. 9, 1915.

THE substance is melted and then sprayed from a nozzle by compressed air or an inert gas, so that it solidifies in a finely divided state.—W. F. F.

**Pulsometer; Automatic— for raising liquids, especially acids**

— J. Cotellet. Fr. Pat. 480,601, Jan. 29, 1916.

LIQUID enters the bottom of a closed vertical vessel through a non-return valve. The top of the vessel is provided with passages communicating with the atmosphere and with a supply of compressed air, the openings being controlled by two vertical glass rods resting on the opposite ends of a pivoted lever at the bottom, so that the rods move simultaneously in opposite directions. A float surrounds the rod controlling the passage to the atmosphere and engages with stops at both ends of the rod. When the float is at the bottom, the atmospheric valve is open and the compressed air valve closed and liquid fills the vessel and raises the float. When the vessel is full, the atmospheric valve is closed and the compressed air valve opened, so that the liquid is forced out through another pipe at the bottom and the float falls. The cycle is then repeated.—W. F. F.

**Furnaces of the tilting type**

— I. Hall, Birmingham. Eng. Pat. 102,844, Feb. 15, 1916. (Appl. No. 2214 of 1916.)

**Impregnating liquids with carbonic acid or other gases; Apparatus for**

— A. A. Pindstoft, Copenhagen. U.S. Pat. 1,209,490, Dec. 19, 1916. Date of appl., Sept. 17, 1914.

SEE Eng. Pat. 19,769 of 1914; this J., 1915, 341

**Recovering solvents; Apparatus for**

— T. Baker, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,209,657, Dec. 26, 1916. Date of appl., Oct. 7, 1912.

SEE Eng. Pat. 10,351 of 1914; this J., 1915, 601

**Spray-producer or atomiser for liquids**

— D. E. Thomas, Morriston, Wales, A. D. Berk, London, and F. J. Morgan, Swansea. U.S. Pat. 1,210,232, Dec. 26, 1916. Date of appl., Dec. 29, 1915.

SEE Eng. Pat. 608 of 1915; this J., 1915, 1132.

**Separating substances of different specific gravities, such as fine coal or ores and the like; Apparatus for**

— J. M. Draper, Manchester. U.S. Pat. 1,210,916, Jan. 2, 1917. Date of appl., Jan. 24, 1916.

SEE Eng. Pat. 1849 of 1915; this J., 1915, 1106.

**Separation of liquids by centrifugal force; Process and apparatus for**

— L. P. Sharples. Fr. Pat. 480,726, Jan. 21, 1916. Under Int. Conv. Apr. 27, 1915.

SEE U.S. Pat. 1,207,230 of 1916; preceding.

**Apparatus for continuous distillation**

— Fr. Pat. 480,553. See XVIII.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Ammonia recovery (from coal gas) and intensive scrubbing.* W. S. Blauvelt. Gas Record, 1916. Gas J., 1917, 137, 63–64.

THE factors operative in washing gas by water or ammonia recovery are considered, and it is pointed out that an equilibrium between ammonia in the gas and in the wash liquor may limit the effectiveness of the process. Washers are classified under four types:—1. Washers in which the gas bubbles through the wash liquor. These may cause considerable back pressure. 2. Tower scrubbers, up which the gas ascends onco meeting a descending stream of wash liquor. Time and area of contact are increased by fillings of coke, tiles, or wooden grids. The cost is relatively low, and back pressure is small. The large size required renders this type unsuitable for large works. 3. Mechanical washers containing moving internal parts which effect good contact between gas and liquor. They are usually multi-stage with gas moving counter to the liquor. 4. "Intensive scrubbers," comprising a number of towers or compartments in series, with grid fillings. The liquor passes repeatedly down each tower before proceeding to the next. Gas and liquor flow in counter currents. Types 3 and 4 are more adapted for use in larger works, and it is considered that the "intensive scrubber" has the advantage over the mechanical washer. In the latter a large portion of the contact surface is always submerged and out of action, in the former it is all in action. Installation costs, and over a long term of years maintenance costs also are lower, and ground space less, while the "intensive scrubber" also works better under overload conditions.—H. J. H.

*Petroleum Supplies Branch of the Ministry of Munitions.*

THE Minister of Munitions has established a Petroleum Branch in the Ministry, to be known as the Munitions Petroleum Supplies Branch, to deal with questions of the provision and distribution of petroleum and similar mineral oils for the purposes of the Ministry of Munitions. Mr. E. Houghton Fry will act as Director of the Branch. In connection with the investigation and development of hitherto unproved home sources of supply of mineral oils, the Minister will have the advantage of the services of Sir Boverton Redwood as Director of Munitions Petroleum Research. Sir Boverton Redwood will have charge of the Research Section, including the development of the British sources of supply. Professor John Cadman, C.M.G., President of the Institute of Petroleum Technologists, has assisted in the organisation of arrangements with the Scottish shale distilleries during the war, with a view to a considerable increase in their output. Sir George Beilby is also assisting in an advisory capacity.

*Gas-oils; Cracking of—in various atmospheres.* R. C. Downing and E. F. Pohlman. Amer. Gas Institute. Gas J., 1917, 137, 24–26.

THE atmospheres used were nitrogen, carbon dioxide, carbon monoxide, hydrogen, methane, blue gas, and a mixture of blue gas with 10 and 20% of steam respectively. The effect of each diluent on candle-power of the gas, candles per gallon, B.Th.U. per gallon, and the production of gas, tar, and carbon was studied. With nitrogen the calorific power of the gas per gallon of oil was 97,000 B.Th.U. at 1350° F. (about 730° C.). This is slightly better than is obtained in atmospheres of carbon monoxide, carbon dioxide, and methane, and lower than in hydrogen or blue gas. The

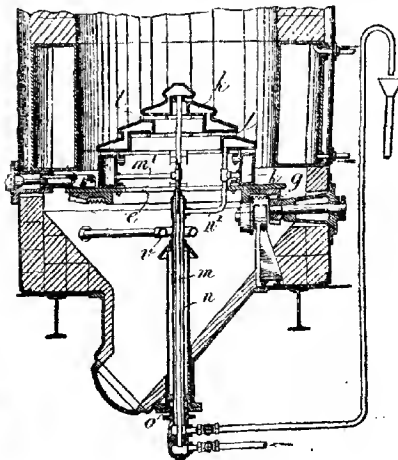
amount of tar and carbon produced was considerable, as is usually the case in inert atmospheres. The yield of oil-gas by weight was rather low. In an atmosphere of carbon dioxide the volume of the gas made was over 60 cu. ft. per gallon of oil, and it contained much hydrogen and methane. The production of carbon was much increased. The reaction  $2CO + C_2H_4 = 4CO + 2H_2$ , probably occurs to a slight extent. With hydrogen, gas of high calorific power tends to be produced, owing to the production of saturated compounds. There is a decrease in the formation of tar and carbon. The production of illuminants is low; apparently some of the unsaturated compounds are hydrogenated. Methane acts very much like an inert atmosphere; much less methane is actually produced from the oil, but the production of hydrogen is larger, due probably to the decomposition of the methane. The effect of blue gas was studied in some detail. The conclusion is reached that no advantage is to be gained over blue gas by the use of any of the above-mentioned atmospheres alone. Blue gas gives much better candle-power results than either hydrogen or carbon monoxide. Tar formation is lowered and the deposition of carbon is about the average. Steam lowers the candle-power and candles per gallon, but raises the B.Th.U. per gallon of oil, possibly due to formation of hydrogen by partial decomposition of the steam. The appearance of the flames when the gas formed in the various atmospheres was burned in a Bray burner is also described. With carbon monoxide great loss of candle-power was noticed, due to the large proportion of carbon monoxide in the finished gas. The B.Th.U. results were the lowest obtained with any atmosphere except from oil used alone with a much longer time of contact. More tar was produced with carbon monoxide than in any other atmosphere. Carbon monoxide moreover seems to prevent the decomposition of the "illuminants." (See also this J., 1916, 917.)—F. G.

*Combustion methods for use in the laboratory.* [Determination of sulphur.] Hewett. See XXXIII.

PATENTS.

*Gas producers.* F. Thuman, London. Eng. Pat. 102,597, Sept. 16, 1916. (Appl. No. 13,173 of 1916.)

THE bottom of the producer is provided with a base-plate, *c*, rotated by bevel gearing, *g*, and carrying a grate having water-circulating channels,



k. Cooling water is supplied by the pipes, *m*, *m*<sup>1</sup>, and withdrawn by the pipes, *n*<sup>1</sup>, *n*. Steam is supplied through the sprayer, *v*, and passes with air to the fire through the openings, *l*. The water pipes are protected from the hot ashes by a surrounding sleeve carried by the stuffing-box, *o*.

—W. F. F.

**Solidified hydrocarbons; Treatment of — for the manufacture of combustible blocks.** A. C. B. Webb. Fr. Pat. 480,347, Nov. 29, 1915. Under Int. Conv., Oct. 30, 1915.

DEHYDRATED calcium sulphate, clay, or earth is mixed with wax, heavy mineral oil, or heavy vegetable oil to form a porous combustible mass which may be ignited by pieces of celluloid or cotton waste.—J. E. C.

**Coal gas; Apparatus for manufacture of —.** The Ritter-Conley Manufacturing Co. Fr. Pat. 480,534, Dec. 6, 1915.

SEVERAL gas retorts are connected to a common stand-pipe, to secure a uniform temperature high enough to fix the lighter hydrocarbons in the gas (about 1000° F., 540° C.). The stand-pipes communicate with short mains, in which the gas is cooled by liquid sprays to about 110° to 125° F. (43° to 52° C.). The gas then passes to the general purification plant. Means of access to the stand-pipes and short mains are provided.

—J. E. C.

**Gas; Manufacture of —.** L. D. Carroll. Fr. Pat. 480,535, Dec. 7, 1915.

IN the manufacture of carburetted water-gas or similar gases, the primary and secondary air are controlled so as to give a constant volume of air per unit of time. The regulation may be effected by governors acting on the source of power driving the air blower and controlled by the differential pressure produced by a Venturi meter, perforated diaphragm, or Pitot tube.

—J. E. C.

**Gas; Manufacture of —.** L. D. Carroll. Fr. Pat. 480,536, Dec. 7, 1915.

IN a gas-producing system in which a gas with increasing content of carbon monoxide is produced, the secondary air is regulated in proportion by a throttle-valve in the air conduit and controlled by the rise and fall of a governor bell. The bell is actuated by a three-way cock alternately connecting the contents with the air conduit and the atmosphere. Means are also provided for counteracting the effect of increased pressure in the air conduit.—J. E. C.

**Coal gas; Apparatus for manufacture of —.** L. D. Carroll. Fr. Pat. 481,095, Mar. 1, 1916.

IN a vertical retort system for the manufacture of coal gas, one of the retorts communicates directly with a producer and supplies the necessary coke. Means are provided for regulating the pressure in this retort and in the producer to prevent the producer gas passing into the coal gas retort.—J. E. C.

**Hydrocarbons; Extraction of — from coal gas.** G. E. Darier. Fr. Pat. 479,997, Jan. 12, 1915.

HYDROCARBONS of the benzene and ethylene series are recovered by cooling coal gas to a temperature low enough to precipitate these bodies in a solid or liquid state. A recuperative system is provided by using two coolers alternately, in one of which the hydrocarbons are precipitated in a solid state, whilst in the other the solid products are liquefied by passing the uncooled gas through the chamber. The solid

products may also be extracted by solvents, the solvents solidifying with difficulty may be introduced prior to cooling. The hydrocarbons may be separated at higher temperatures by introducing porous substances, such as animal charcoal, coconut fibre, etc., whilst benzene compounds may be selectively separated, leaving ethylene compounds in the coal gas.—J. E. C.

**Gaseous mixtures; Proportioning of —.** A. C. Ionides, jun., London. Eng. Pat. 17,670, Dec. 17, 1915.

THE proportions of the constituents of a self-burning gaseous mixture are adjusted by observation of the lighting effect produced by combustion with an incandescent mantle under regulated pressure conditions. The illumination may be compared with a standard source of light or with a guide light using a mixture of known proportions.—J. E. C.

**Producer-gas; Process for producing a diffused flame with —.** H. L. Doherty, New York. U.S. Pat. 1,206,821, Dec. 5, 1916. Date of appl. May 15, 1911. Renewed Mar. 11, 1916.

THE flame is regulated to produce a uniform heating effect by mixing from 5 to 100% of combustion gases with the producer gas and burning it in a furnace chamber with preheated air, so that a slow burning flame of moderate temperature fills the chamber.—W. F. F.

**Hydrocarbon oils; Method of dehydrating and refining —.** E. I. Dyer, Oakland, Cal. U.S. Pat. 1,207,381, Dec. 5, 1916. Date of appl. Mar. 5, 1913.

THE oil is made to flow in a thin layer over heated surfaces in a closed vessel, and, after removal of the lighter vaporised portions, it is conducted, without cooling, into a second closed vessel, where it is again made to flow circuitously over heated surfaces at a higher or lower temperature and pressure than in the first vessel. Outside heating is used for one of the vessels, and the vapours generated therein are conducted through the other vessel as a heating medium for the oil. The oil in each vessel is agitated during the heating, and means are provided for condensing the vapours which have been used for heating one of the vessels.—O. A. M.

**Petroleum; Apparatus for converting heavy products obtained from —.** Process for the conversion of heavy products of petroleum. P. Roth and M. E. Venturino, Buenos Aires, Argentina. U.S. Pats. (A) 1,208,214 and (B) 1,208,575, Dec. 12, 1916. Date of appl. Oct. 16, 1915.

(A) PETROLEUM products are successively heated in a series of connected boilers, each of which is provided with means for discharging and condensing the vapours formed in the distillation. The heavy or middle hydrocarbons thus obtained are separated from water and impurities, and are sprayed under pressure into the vapour space of the lowest or converting boiler, which is also connected with a condenser to receive the vapours formed in the process. (B) Petroleum residues, asphalt, oils, or crude petroleum are sprayed into the vapour space of a still above a layer of similar material which has been heated to a temperature above that required to "crack" the heavy hydrocarbon or crude petroleum to be treated, whereby partial vaporisation of the sprayed liquid is effected, while the greater proportion of the volatilisation and cracking occur when the liquids come into contact. The resulting vapours are condensed.—O. A. M.

*Petroleum oils; Process and apparatus for transforming heavy—into light oils.* Simplex Refining Co. Fr. Pat. 480,147, Nov. 8, 1915.

HEAVY petroleum oil is made to circulate, without pressure, in a continuous cycle, e.g., through serpentine tubes, which are heated to the "cracking" point of the oil, and means are provided for the continuous withdrawal of the resulting vapour and the addition of fresh oil to keep the circulating oil at a constant volume. A portion of the condensed light vapour is injected into the residue when it returns to the heating vessel. Advantages claimed are the process is regularity in working, and the prevention of overheating and formation of carbonaceous and viscous deposits within the tubes.—C. A. M.

*Chemical reactions; Producing—in a single [gaseous] phase. [Cracking hydrocarbons.]* Synthetic Hydro-Carbon Co. Fr. Pat. 480,653, Jan. 11, 1916.

A VERTICAL tube of refractory material heated electrically by a surrounding coil, is provided at the top with a plug of loose refractory material through which liquid hydrocarbon is supplied to be gasified and then cracked. The products, which vary with the temperature, pressure, and the rate at which the gasified material flows through the heated zone, pass through a condenser to a liquid-collecting vessel having a pipe leading to a pump for withdrawing the gas at the top. Light hydrocarbon oils are produced at temperatures of 600°–900° C. and pressures about 6 atmospheres. Permanent gases are produced in large proportion at a pressure of about 0.28 kilo. per sq. cm.—W. F. F.

*Motor spirit suitable for high-speed internal combustion motor engines; Process of producing—.* Hall Motor Fuel, Ltd. Fr. Pat. 481,066, Feb. 25, 1916.

THE fraction of kerosene oil boiling up to about 220° C., which must be practically free from petroleum spirit, is forced through very narrow interstices at a high pressure (70 to 200 kilos. or more, per sq. cm.), in the presence of hydrogen or a hydrocarbon gas, and at a temperature not exceeding 100° to 120° C. or that of the initial boiling point of the liquid. The resulting product burns much more rapidly than kerosene oil in motor engines.—C. A. M.

*Petroleum or other hydrocarbons; Apparatus for the continuous fractionation and dephlegmation of the products of the distillation of— or of a mixture of volatile liquids, including products of the distillation of shale, coal, or both.* H. L. Allan. Fr. Pat. 481,134, Mar. 8, 1916.

THE vapour from the first distillation is made to pass downwards through vertical tubes of a dephlegmator, which are exposed to the air, and may also be cooled by water which trickles over them and is collected in a tray. The condensed liquid which collects in the vessel below the tubes may be redistilled by means of a steam coil or the like, and the resulting vapour passed upwards through other vertical tubes into a box at the top connecting the series of tubes, and thence to a water-condenser. In this process the latent heat of the condensed fractions is utilised in the redistillation of the portions of lower boiling point. The fractions collected at each stage are of uniform quality.—C. A. M.

*Coal gas retorts; Charging machines for use in operating—.* A. E. White, London. From Riter-Conley Manufacturing Co., Leedsdale, Pa., U.S.A. Eng. Pat. 102,839, Jan. 19, 1916. (Appl. No. 857 of 1916.)

*Coke-oven.* E. Hinselmann, Essen, Germany. U.S. Pat. 1,208,781, Dec. 19, 1916. Date of appl., Nov. 24, 1914.

SEE Ger. Pat. 289,082 of 1913; this J., 1916, 527.

*Vertical retorts; Discharging—.* A. McD. Duckham, Ashtead. U.S. Pat. 1,211,301, Jan. 2, 1917. Date of appl., May 19, 1915.

SEE Eng. Pat. 17,068 of 1914; this J., 1915, 215.

*Gas; Burners and methods of burning—.* W. K. Twomey, Toledo, Ohio, U.S.A. Eng. Pat. 18,092, Dec. 28, 1915.

SEE U.S. Pat. 1,167,447 of 1916; this J., 1916, 298.

*Gas producers of the Mond type; Method of working—.* J. B. Chadwick and P. Hollinshead, Runcorn, Assignors to The Salt Union, Ltd., Liverpool. U.S. Pat. 1,210,598, Jan. 2, 1917. Date of appl., July 12, 1912.

SEE Eng. Pat. 16,168 of 1911; this J., 1912, 975.

*Gas producers.* S. Glover and J. West. Fr. Pat. 480,249, Nov. 10, 1915.

SEE Eng. Pat. 5315 of 1915; this J., 1916, 297.

*Coal gas; Process and apparatus for manufacture of—.* A. Waddell. Fr. Pat. 481,050, Feb. 24, 1916. Under Int. Conv., Apr. 14 and Oct. 6, 1915.

SEE Eng. Pats. 5811, 14,142, and 14,143 of 1915; this J., 1916, 625, 1147.

*Method of making [graphite] lubricants.* U.S. Pat. 1,207,843. See I.

*Method of producing granular carbon from [oil] gas refuse.* U.S. Pat. 1,207,604. See IIb.

*Process for the production of ammonia from coke.* Eng. Pat. 101,154. See VII.

*Apparatus for the analysis of gas.* Fr. Pat. 480,661. See XXIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Carbon from [oil-] gas refuse; Method of producing granular—.* R. D. Pike, San Francisco, Cal. U.S. Pat. 1,207,694, Dec. 5, 1916. Date of appl., Apr. 1, 1916.

LAMPBLACK from the refuse of oil-gas manufacture is heated to a relatively low temperature to expel water, and is then mixed with oil-gas tar to form a semi-plastic mass which is pressed into cakes. The cakes are heated to a high temperature to expel all volatile products and the residue finely ground and subjected to magnetic separation.—W. F. F.

*Arc light carbons, and method of manufacturing the same.* R. D. Pike, Richmond, Cal., U.S.A. Eng. Pat. 102,645, Feb. 21, 1916. (Appl. No. 2541 of 1916.)

IN the production of arc light carbons from the so-called lampblack refuse from the manufacture of illuminating gas from crude petroleum, the tar and soluble ash are removed from the refuse by washing. Volatile hydrocarbons are then removed by roasting, and the resulting product ground and incorporated with a suitable flux (such as boric acid) to produce a plastic body. Carbons are formed from the plastic mass in the usual manner.—J. E. O.

*Arc device; Inclosed — and method of operating the same.* G. M. J. Mackay, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,208,597, Dec. 12, 1916. Date of appl., Oct. 30, 1915.

A REFRACTORY cathode, such as tungsten, with a melting point exceeding 2000° C., is adapted to co-operate at incandescence with an anode within a sealed envelope, and the latter is filled with a monatomic gas, such as argon, with a pressure, at the operating temperature, of several millimetres of mercury, the gas being inert with respect to the electrodes and suppressing the electrical disintegration of the cathode. A drying material, inactive with respect to argon, is employed to remove the water vapour emitted from the electrodes and the walls of the envelope. The drying material is liberated in the form of vapour, by the operation of the arc, and forms compounds of negligibly low vapour pressure by combination with the water vapour.—B. N.

[*Tungsten*] *metal-filament manufacture.* O. Oberländer, London, Assignor to General Electric Co. U.S. Pat. 1,208,629, Dec. 12, 1916. Date of appl., Nov. 27, 1911.

A PLASTIC mass, for the manufacture of tungsten filaments, is formed by treating tungsten hexachloride with methylated ether, and then evaporating the resulting product to a paste of suitable consistency for further treatment.—B. N.

*Electric incandescence bodies; Process for manufacturing —.* C. O. Bastian, London. U.S. Pat. 1,209,247, Dec. 19, 1916. Date of appl., Sept. 24, 1914.

SEE Eng. Pat. 22,331 of 1913; this J., 1914, 1197.

*Electron-emitting cathodes and process of manufacturing the same.* A. McL. Nicolson, New York, and E. C. Hull, Montclair, N.J., Assignors to Western Electric Co., Inc. U.S. Pat. 1,209,324, Dec. 19, 1916. Date of appl., Dec. 26, 1914.

SEE Eng. Pat. 17,580 of 1915; this J., 1916, 828.

*Carbons for arc lamps, and process of making them.* R. D. Pike. Fr. Pat. 481,074, Feb. 20, 1916.

SEE Eng. Pat. 162,645 of 1916; preceding.

*Process and [electric] fusion furnace for the manufacture of coherent ductile tungsten or other metal [for incandescence filaments].* Fr. Pat. 480,819. See X.

### III.—TAR AND TAR PRODUCTS.

*Aromatic base oil; Cracking of —. The temperature factor at constant rate under pressure.* G. Engloff and R. J. Moore. J. Ind. Eng. Chem., 1917, 9, 40—42.

FOR the production of benzene and toluene from an aromatic base oil, the best results were obtained with solvent naphtha. The oil was passed through a steel tube, 11½ ft. long by 8 in. in diameter, at a pressure of 11 atmos. and at the rate of 15 galls. per hour, the temperatures used being 500°, 550°, 600°, 650°, 700°, 750°, and 800°C. In the recovered oil the maximum yield for benzene was 42.5% at 800° C., and for toluene 39.9% at 750° C.; or, calculated on the basis of the solvent naphtha used, 15.9% for benzene and 20.6% for toluene. The following table shows the maximum yields obtained under comparable conditions from a paraffin base oil and a naphthene base oil:—

	Paraffin oil		Naphthene oil		Aromatic oil	
	% by vol.	°C.	% by vol.	°C.	% by vol.	°C.
Benzene ..	6.6	750	7.2	650	15.9	800
Toluene ....	4.5	650	6.0	650	20.6	750

The course of the decomposition effected by the cracking of the solvent naphtha is of the same kind as the reaction produced by aluminium chloride on alkyl derivatives of benzene, which are converted successively into lower alkyl derivatives, toluene, and benzene (Ber, 1884, 17, 2816; this J., 1885, 270). The yield of recovered oil decreased from 100% at 500° C. to 98% at 650° and to 37.3% at 800° C. The greatest decomposition (25%) took place between 700° and 750° C. The sp.gr. of the recovered oils increased from 0.868 at 500° C. to 0.989 at 800° C., which indicated the formation of polycyclic aromatic hydrocarbons of high molecular weight.

—C. A. M.

*Trinitrobenzene; Additive compounds of —.* J. J. Sudborough. Chem. Soc. Trans., 1916, 109, 1339—1348.

POLY-NITRO compounds of the aromatic series possess the property of forming additive compounds with aromatic hydrocarbons, amines, phenols, phenolic ethers, and sulphur compounds. A large number of additive derivatives of *s*-trinitrobenzene have been prepared and analysed. In the majority of cases the compound is obtained by mixing theoretical proportions of the components and crystallising from a suitable solvent, usually alcohol, but occasionally benzene. In other cases the compound was only obtainable by using an excess of the hydrocarbon or base over the amount calculated to form the trinitrobenzene derivative. As a rule the colour of the additive compounds with hydrocarbons is not so marked as that of the corresponding amino- or hydroxy-derivatives; the colour of the hydrocarbon additive compounds is usually yellow, but anthracene gives a bright red compound. It would appear that the formation of these compounds is due to latent valencies of the nitro-groups and of the aromatic residues of the hydrocarbon; the hydroxy-, alkyloxy-, amino-, and alkylamino-groups act mainly as auxochromes and tend also to increase the stability. As regards the combining proportions, it would appear that the number of trinitrobenzene molecules combining varies with the number of independent aromatic nuclei in the other component; in this connection, however, a condensed system of several benzene or benzene and heterocyclic rings counts as a single nucleus. Compounds in equimolecular proportions are formed with naphthalene, anthracene, phenanthrene, aniline, etc. Compounds of two mols. of trinitrobenzene and one mol. of the substance are formed with stilbene, diphenylamine, etc. There are, however, exceptions to the rule, and several substances form two types of additive compounds containing one and two mols. of trinitrobenzene respectively. Quite a number of substances containing two or more independent aromatic nuclei combine in equimolecular proportions; in a few cases one mol. of trinitrobenzene combines with two of the substance.

—J. F. B.

*s*-Trinitrobenzene; Additive compounds of — with amino-derivatives of complex aromatic hydrocarbons. S. T. Cadre and J. J. Sudborough. Chem. Soc. Trans., 1916, 109, 1349—1354.

As a rule the additive compounds (see preceding

abstract) obtained from naphthalene derivatives are more stable than those derived from corresponding benzene compounds. A series of compounds has been prepared from amino derivatives of phenanthrene, anthracene, acenaphthene, and fluorene. With the exception of aminofluorene all these compounds were formed by the union of equimolecular proportions of trinitrobenzene and the base. In this series, the colours of the additive compounds are relatively deep, being, as a rule, either black or deep brown, whereas those of the naphthalene series are mainly orange-yellow or red. The compounds are comparatively stable and can be crystallised from most solvents, but determinations of molecular weights showed that, in dilute benzene solution, dissociation into the components is almost complete. These compounds, on the whole, resemble the additive compounds from the naphthylamines, but several of them appear to be incapable of yielding acetyl derivatives.—J. F. B.

*Benzoyl chloride; Catalytic decomposition of* —. A. Mailhe and P. de Godon. Bull. Soc. Chim., 1916, 19, 449—452.

BENZOYL chloride when passed with a current of hydrogen over finely divided nickel at 270°—280°C. furnishes to the extent of about 50% a mixture of benzene and toluene, and about 40% of diphenyl. The mechanism of the formation of the latter is obscure, but it appears to be conditioned by the formation of nickel chloride on the surface of the catalyst, and is not suppressed even when an excess of hydrogen is employed. With copper as catalyst benzoyl chloride is decomposed up to about 40% into benzoic anhydride, the remainder being unchanged. A similar conversion occurs at 420°—450°C. with the chlorides of barium and thorium, with simultaneous deposition of carbon on the catalyst, and formation of carbon monoxide and hydrogen chloride. The manner of formation of the anhydride remains uncertain.—G. F. M.

*Friedel-Crafts reaction; Observations on the* —. [Decomposition of polyalkylbenzenes.] E. Bedtker and O. M. Halse. Bull. Soc. Chim., 1916, 19, 444—449.

A REVERSAL of the Friedel-Crafts reaction can in general be brought about with more or less facility by the action of aluminium chloride on the alkylbenzene in presence of a large excess of benzene. In the case of the xylenes alone, this reaction does not succeed, but when polyethyl-, isopropyl-, butyl-, and amylbenzenes were boiled with aluminium chloride in presence of ten times their weight of benzene, quantities of the monoalkylbenzene, varying in each case were formed according to the scheme:—



Of the greatest interest however was the formation in excellent yield of toluene and cumene from cymene, which is available in large quantities as a by-product of the sulphite-cellulose process. Ninety grms. of cymene in 900 grms. of benzene gave, on boiling for 10 hours with 4.5 grms. of aluminium chloride, a yield of 44 grms. of toluene and 68 grms. of cumene, corresponding to 80% of that theoretically possible, leaving a residue of only 3—4 grms. of gummy material. The polyhalogen derivatives of benzene could not be decomposed by the action of aluminium chloride and benzene in this manner.—G. F. M.

*Determination of naphthalene by picric acid.* Knublauch. See IIA.

*Combustion methods for use in the laboratory.* [Determination of sulphur.] Hewett. See XXIII.

## PATENTS.

*Aromatic hydrocarbons; Improving the colour and odour of crude* —. J. M. Weiss, Assignor to The Barrett Co., New York. U.S. Pat. 1,206,962, Dec. 5, 1916. Date of appl., Jan. 22, 1913.

THE oil to be purified is intimately mixed with an aqueous solution of a metallic salt, such as copper sulphate, in the proportion of 1 to 3 lb. of the hydrated salt to 100 galls. of oil, and the reaction allowed to proceed at a temperature below the point at which distillation of the volatile ingredients of the mixture occurs. The mixture is settled, the aqueous liquor drawn off, and the remaining oil distilled.—B. N.

*Sulphonic acids; Process of manufacturing sodium salts of* —. A. P. Sachs and O. Byron, Carnegie, Pa. U.S. Pat. 1,207,798, Dec. 12, 1916. Date of appl., Apr. 6, 1916.

A MIXTURE of a sulphonic acid forming a water-soluble calcium salt, sulphuric acid, and water is neutralised with lime to obtain a mixture of calcium sulphate and the calcium salt of the sulphonic acid. This mixture is agitated, without previous filtration, with sodium sulphate, and then filtered.—F. W. A.

*Aromatic amino-[hydroxy] compounds; [Electrolytic] manufacture of* —. O. Imray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 18,081, Dec. 28, 1915.

IN the electrolytic reduction of aromatic nitro-compounds, the formation of the amino-hydroxy compound is favoured and that of the amine reduced, by the use of a cathode of two or more metals. The cathode is obtained either by the use of a suitable alloy, the surface of which remains of practically constant composition during the reaction, or by making the cathode of one metal and placing the other, either as such or as salt, in the electrolyte, or by using an indifferent substance such as carbon and depositing the metals thereon during the electrolysis. It is possible to reduce the amount of acid to very little more than is necessary to combine with the base which is formed. Details are given of the reduction of nitrobenzene using a copper cathode, with lead, or lead and arsenic, in the electrolyte, and with a lead cathode with bismuth in the electrolyte. Other suitable combinations of metals are copper with mercury, and copper with tin and arsenic. A plain lead cathode under these conditions would give *p*-aminophenol and aniline in the proportion of about 2 to 3, while this method gives them in the proportion of 5 or 6 to 1.—B. V. S.

*Monochlorobenzene; Manufacture of* —. G. Coutagne. Fr. Pat. 480,151, Feb. 17, 1915.

MONOCHLOROBENZENE is formed by the action of chlorine on benzene, in the presence of a catalyst such as iron, the process being so conducted that strong chlorine vapour comes into contact only with fresh benzene. A stream of benzene flows through a vertical series of basins exposed to a stream of chlorine passing in the same direction as the benzene. The formation of higher chlorine compounds is further reduced by mixing with the chlorine vapour hydrochloric acid previously formed by the reaction. Cooling appliances are used for maintaining the benzene at a temperature of 15°C.—B. V. S.

*Binding compound [from coal tar, etc.] and process of making same.* A. C. Evans, Hampton Wick, and P. J. Mitchell, London. U.S. Pat. 1,200,692, Dec. 26, 1916. Date of appl., Sept. 7, 1915.

SEE Eng. Pat. 10,933 of 1914; this J., 1915, 899.



*Dephlegmator* [for benzol stills]. Eng. Pat. 17,567.  
See I.

*Apparatus for the continuous fractionation and dephlegmation of the products of the distillation of petroleum and other hydrocarbons.* Fr. Pat. 481,134. See IIA.

#### IV.—COLOURING MATTERS AND DYES.

*Indigo in Bihar; Third report on the improvement of —.* A. and G. L. C. Howard. Bull. No. 67, 1916. Agric. Research Inst., Pusa. (See also this J., 1915, 952.)

THE indigo plant can assimilate nitrogen either through the agency of *B. radiclecola* present in its root nodules or from nitrates present in the soil. The nodules are numerous and functionally very active in poor, well-aerated soils, but their development and activity are arrested and inhibited in rich ground, the nitrogen in this case being derived from nitrate. The formation of indican is dependent upon the activity of the nodules and is greatest in the high-lying areas; in the rich low-lying swamp tracts and in fields treated with *seeth*, the nitrate cycle predominates, the plants yield heavy leaf crops, but their indican content is low and they afford only a dye of inferior quality. The indican may be regarded as representing the difference between the total nitrogen assimilated and that consumed in growth. The essence of successful indigo cultivation is adequate soil-aeration. Saltpetre soils dressed with potsheds (*thikra*) or brick-refuse (*surkhi* and *roras*) grow remarkably fine indigo, and the value of adding such aerating agents to other soils is now being investigated; so far, it has been ascertained that they are best applied in comparatively small fragments. All the evidence to hand points to the conclusion that wilting is a starvation effect due to inhibited development of the nodules and fine roots; it cannot well be cured, but it may be prevented by cultivating early plants which grow rapidly and which have shallow roots, all weak, late-flowering plants being exterminated, and by adequate drainage. The introduction of new seed from Java would be useless, as the cultivation in that island is now in the hands of natives who do not select the seed carefully; they grow chiefly deep-rooting, late types. The Java plant, however, does not breed true, and if the early, quick-growing forms were selected and cultivated continuously, the old type of Java plant which formerly flourished in Bihar could be recovered. Both Javan and Sumatran varieties can be cultivated during the hot season, thorough weeding and the formation of a surface mulch of several inches being effected with the Canadian lever-harrow. When the first crop is gathered, a branch should be left to maintain the transpiration current, thereby causing the quick growth of shoots for a second cut; after the latter, the plants should be dug out and the land prepared for *rabi* crops, as it does not pay to grow the old stumps for a seed crop. The value of a good drainage system cannot be over-emphasised: an improved system for northern Bihar is a *sine qua non* of the re-establishment of the indigo industry. A special seed crop, well spaced, should be sown early in August and kept for leaf afterwards. Should floods intervene, the soil must be aerated by deep cultivation, and the application of *thikra* and the provision of sub-soil drainage would also assist. Storage of seed is also to be recommended. Attempts to grow Sumatran indigo for seed have not been encouraging, as although the seed was good in quality the yield was small: possibly selection might lead to improvement. Java indigo

from Natal is unsuitable for cultivation in Bihar. The Java plants are fertilised by bees, and cross-pollination is the rule. Artificial self-fertilisation is difficult, and leads to degeneration. The mixed types composing the ordinary crops vary greatly, the late slow-growing kinds yielding the most indican. The chief means of improvement consists in controlling natural crossing. To this end, the seeds of a large number of good, early plants are collected and sown, the best plants are picked out and the weakest removed. The seeds of the former are mixed and sown on a large scale, and again the undesirable individuals are eradicated. Alternatively, the selection may be started from a single plant. Improvement by selection on the basis of indican content would be difficult and probably unsuccessful in Bihar, for those plants which contain most are late, deep-rooting sorts and are liable to wilt. Attempts to improve Sumatran indigo from Madras by selection did not succeed, but the possibility of crossing a high-yielding Java type with a good strain of Cawnpore Sumatran needs investigation. As regards indirect means of improvement, Java indigo might be rotated with *rakar* (rice), as this tends to conserve the soil organic matter and to open up the sub-soil: the rice is also improved by the rotation. The value of wheat as a cover crop was demonstrated by a very successful test with the rapid-growing Pusa 4 wheat: the flour produced was of the finest quality and, after the stubbles had been thoroughly harrowed, the next crop of indigo developed well.—E. H. T.

*Indigo.* Rept. Agric. Res. Inst., Pusa, 1915-16.  
94—96.

THE success or failure of indigo manufacture depends largely upon the nature of the bacterial inhabitants of the steeping vats. Two distinct types of fermentation have been identified, one involving the evolution of nitrogen (usually in large excess) and carbon dioxide, and the other producing hydrogen, nitrogen, and carbon dioxide in about equal proportions. These fermentations do not give rise to methane. Numerous species of bacteria have been isolated and some of them have been classified definitely as beneficial or deleterious. The presence of one bacterium has been correlated with the unfortunate production of "green vat" which occurs sometimes in the "beating" or oxidising process. It is hoped that the manufacture may be improved by artificially controlling the bacteria in the vat.—E. H. T.

#### *Indian indigo crop.*

THE final general memorandum on the 1916-17 indigo crop, issued by the Department of Statistics, India, and based upon reports received from provinces containing practically the whole area under indigo in British India, states that the total area is estimated at 756,400 acres, which is 114% in excess of the acreage (353,100 acres) of last year. All the provinces show an increase in the area sown, the largest increases being in the United Provinces and in the Punjab. The total yield of dye is estimated at 95,500 cwt., as against 55,100 cwt., last year, or an increase of 73%. The present estimate of yield, as against the final figures of last year, shows an increase of 267% in the Punjab, 252% in the United Provinces, 117% in Bombay and Sind, 53% in Bihar and Orissa and 46% in Madras. The season has not, on the whole, been favourable for the crop except in the Punjab and Bombay and Sind. Heavy rainfall and floods adversely affected the crop in the United Provinces and in parts of Bihar and Orissa and Madras. The exports by sea to foreign countries in each of the last five years have been (in cwt.): 1911-12, 19,155; 1912-13, 11,857; 1913-14, 10,939; 1914-15, 17,142; 1915-16,



41,942 cwt. The imports of synthetic and natural indigo into the United Kingdom in the last five calendar years are stated below. Of the total amount of natural indigo (25,157 cwt.) thus imported 1915, 22,322 cwt. was imported from India, 824 cwt. from Java, and the rest from other countries.

	Synthetic indigo.	Indigo.	Total.
	cwt.	cwt.	cwt.
1911 .....	24,237	4,917	29,204
1912 .....	28,302	7,073	35,375
1913 .....	23,889	4,174	28,063
1914 .....	15,517	5,314	20,831
1915 .....	6,542	25,157	31,699
1915 (10 months) .....	—	28,245	28,245

*Flavine and Brilliant Green: new and powerful antiseptics.* Browning and others. See XX.

*Preparation of dyes for the colour sensitising of photographic plates.* Harrison and Bottomley. See XXI.

*Production of dyes for colour-sensitising [photographic plates].* Pope. See XXI.

#### PATENTS.

*Anthraquinone dyes and process of making same.* M. H. Isler, Mannheim, Assignor to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,207,762, Dec. 12, 1916. Date of appl., Feb. 4, 1913.

An oxidation product of dibenzanthrone is heated with *p*-toluidine and boric acid. The products dye cotton from a blue hydrosulphite vat brilliant olive-green shades of excellent fastness to soap and very good fastness to chlorine.—F. W. A.

*Sulphur dyes.* W. Herzberg and O. Scharfenberg, Assignors to Actienges. für Anilinfabr., Berlin. U.S. Pat. 1,209,580, Dec. 19, 1916. Date of appl., Mar. 22, 1913.

See Fr. Pat. 432,440 of 1911; this J., 1912, 119.

*azo] dyestuffs; Orange to red—able to be chromated, and process of making same.* C. Jagerspacher, Assignor to Soc. of Chem. Ind. in Basle, Switzerland. U.S. Pat. 1,210,808, Jan. 2, 1917. Date of appl., Dec. 31, 1915.

See Eng. Pat. 16,916 of 1915; this J., 1917, 78.

*Process for utilising osier-bark.* Fr. Pat. 480,637. See V.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Cotton stalks; Paper pulp from —.* W. B. Nanson. Paper-making, 1916, 35, 371—374.

It has long been known that an excellent fibre suitable for paper pulp or for spinning purposes can be produced from the mature stalks of the cotton plant, although the matter has not been developed beyond the experimental stage. The southern States of America produce 52 million tons of cotton stalks annually, sufficient for 24 million tons of bleached fibre, but the collection of these for industrial utilisation would require some organisation. So far as laboratory trials go, it would appear that the stalks should be chopped in a fodder-cutter into pieces from 2.5—3 ins. long; such a machine would chop 2 tons per hour. The material would then be conveyed to digesters holding 5000—7000 lb. of stock and digested with 1000—5000 galls. of caustic soda liquor at 7°—10° B. (sp. gr. 1.052—1.072) for 10—12 hours;

it is most advantageous to employ a high steam pressure for the digestion, e.g., up to 120 lb. per sq. in. The treatment of the digested pulp would be similar to that of wood pulp. If the fibre is to be treated for spinning purposes, the digested stalks should be washed and passed between steel rollers, such as are employed in cottonseed oil mills, to crush the stalks and separate the fibres. The cotton-stalk pulp is particularly adapted for the permanganate bleaching process, the material being steeped overnight in a solution of sodium permanganate of 0.5°—2° B. (sp. gr. 1.003—1.014); this liquor should be run off and the stock boiled, without washing, in a 5% solution of soda ash for 6—8 hours; the permanganate treatment may be repeated, and the material steeped in sodium carbonate solution before being finally cleared in a bath of sulphurous and sulphuric acids.—J. F. B.

*Friedel-Crafts reaction.* [Decomposition of poly-alkylbenzenes.] Beädtker and Halse. See III.

#### PATENTS.

*Wool and other fibrous materials or the like; Machines for washing —.* T. Rhodes, Shipley, Yorks. Eng. Pat. 17,643, Dec. 17, 1916.

In a wool-washing machine in which the material is carried forward through a trough by means of prongs, inverted funnels closed at the top are attached at their small ends to the cross-members of the rake frame, being either secured to an extension at the base of the prongs or substituted for prongs; valves may be provided in the funnels to regulate the pressure of the enclosed air. The vertical reciprocating motion of the funnels agitates the liquid and air, forcing and sucking them through the goods.—J. F. B.

*Artificial silk, yarns, tow, rope, etc. [from esparto]; Manufacture of —.* L. N. Trigos. Fr. Pat. 480,606, Jan. 3, 1916.

ESPARTO is treated in a boiler with steam or boiling water in presence of 1—6% of its weight of soda or potash until the material becomes soft and pliable. It is then treated in a pressing machine which squeezes out the excess of liquid and separates the fibres from each other, converting the material into a kind of tow which is washed, dried, and combed. The fibre may be bleached with sulphur dioxide and applied to various textile uses.—J. F. B.

*Osier-bark; Process for utilising —.* N. V. Stoom-Touwslagerij en Spinncrij, voorheen onder J. M. Van der Lely. Fr. Pat. 480,637, Jan. 8, 1916. Under Int. Conv., Nov. 16, 1915.

OSIER bark is boiled in a solution of sodium carbonate until the bast tissues are separated into filaments; the latter are washed and dried, with or without bleaching, then combed and spun like hemp or employed as a stuffing for mattresses, cushions, etc. The solution, which contains the tannic acid of the bark, may be employed for tanning or mordanting, and a brown colouring matter may be prepared therefrom.—J. F. B.

*Cellulose and its derivatives; Manufacture of —, and application of the same in various chemical and other industries.* A. C. Vournasos, Athens. Eng. Pat. 17,318, July 22, 1914.

CELLULOSE of fine staple and superior in absorbent power to ordinary cotton may be produced from the fibres of *Hibiscus cannabinus* and its various species of the order *Malvaceae*. The green stems are retted, e.g., by immersion in running water for 30—40 days, and the fibres are opened, scutched, and combed, bleached for 30—40 hours in a solution yielding chlorine, e.g., of potassium hypochlorite, washed free from chlorine, and finally dried in sunlight.—F. Sp.

*Cellulose; Process of manufacture of—and utilization of the waste liquors of the manufacture.* O. Harnist. Fr. Pat. 477,895, July 6, 1914.

RAW cellulose material is treated with boiling water to expel the air; the vessel is then closed and connected with a supply of ammonia gas; a quantity of ammonia is introduced equivalent to one-sixth of that necessary to form the ammonium sulphite it is desired to have in the final exhausted lye, which is generally 2–5% of the weight of the liquor. Ebullition is maintained by indirect heating for  $\frac{1}{2}$ –1 hour under a pressure of 2–4 atmos. At this stage a preliminary removal of organic matter may be effected, e.g., recovery of tannins, resins, etc., by precipitation, on neutralising with sulphur dioxide. After separating the precipitate, the liquor is treated with a further quantity of sulphur dioxide equal to two-sixths of the desired total and digestion of the material continued for 2 hours at 2–4 atmos. The liquor is gradually strengthened with five-sixths of the total ammonia and after one hour this is neutralised with three-sixths of the total sulphur dioxide, and digestion completed in 4–6 hours at 6–8 atmos. The waste liquors are treated for recovery of the reagents or for use as a fertiliser.—J. F. B.

*Luminous [cellulose] product.* Soc. Anon. do Traitement Chimiques. Fr. Pat. 480,230, Nov. 13, 1915.

A FINELY powdered luminous (phosphorescent) substance is suspended in a "collodion," either nitrocellulose, cellulose acetate, or viscose, etc., in appropriate solvents, and the solution is converted into the form of sheets or threads, which can be used for the manufacture of luminous devices. As luminous substances, phosphorescent zinc sulphide, calcium sulphide, barium platino-cyanide, etc., may be employed, either with or without the addition of a salt of radium, mesothorium, or other radioactive substance.—E. W. L.

*Pulp; Process of treating.* G. Moore, Joplin, Mo. U.S. Pat. 1,207,978, Dec. 12, 1916. Date of appl., Jan. 23, 1915.

FOR recovering paper pulp from water containing the particles in suspension, a filter cell is submerged in the water and the latter is passed through the submerged cell until a layer of pulp solids has been deposited thereon; the coated cell is exposed to the atmosphere and air is drawn through the coating into the cell; the cell is again submerged and the layer of pulp discharged from it. The paper pulp suspended in the water may be thickened by the means described and the thickened pulp delivered to a body of similar pulp from which a cake is formed on a screen by a difference in pressure on opposite sides; the cake thus formed is moved into the atmosphere, held in position and drained on the screen by the difference in pressure, and discharged from the screen beyond the body of thickened pulp by reversing the difference of pressure. Water containing pulp in suspension may be treated to thicken the pulp and then aerated to cause the pulp to float by means of bubbles.—J. F. B.

*Waterproofed material and method of producing same.* A. O. Tate, Montreal, Assignor to Tate Electrolytic Waterproofing Co., Inc., New York. U.S. Pat. 1,208,400, Dec. 12, 1916. Date of appl., Dec. 31, 1914.

FIBROUS material is impregnated with a saponaceous liquid and a metallic sulphate, e.g., aluminium sulphate, and then subjected to the action of an electric current passed between electrodes one of which is soluble, whereby a metallic "palmitate" and metallic hydroxide are electrolytically incorporated in the fibres, and co-act therein to make them non-capillary.—J. F. B.

*Paper and the like; Manufacture [sizing] of—* C. W. Fish, Rawcliffe, Yorks. Eng. Pat. 16,742, Nov. 27, 1915.

PAPER pulp is treated with resin size and 0.5–1.0% of an aluminium salt calculated on the dry weight of pulp. When the mixture is thoroughly incorporated, nitre cake is added in quantity about equal to the aluminium sulphate which would be required if the nitre cake were not employed. The order of adding the size and aluminium salt is immaterial, but it is essential that the nitre cake or sodium bisulphate be added last.—J. F. B.

*Wool and other textile fibres and textile or other materials; Machines for washing.* F. B. Petric. Fr. Pat. 480,412, Dec. 8, 1915. Under Int. Conv., Dec. 10, 1914.

SEE Eng. Pats. 23,837 and 23,838 of 1914; this J., 1916, 172.

*Artificial silk; Machine for spinning.* H. de Chardonnet, Paris. U.S. Pat. 1,209,133, Dec. 19, 1916. Date of appl., July 16, 1915.

SEE Eng. Pat. 10,857 of 1915; this J., 1916, 530.

*Sizing paper; Process of—* R. Clavel, Basel, Switzerland. U.S. Pat. 1,211,288, Jan. 2, 1917. Date of appl., Aug. 12, 1916.

SEE Eng. Pat. 101,855 of 1916; this J., 1916, 1215.

*Paper and paper pulp; Process for treatment of—by means of foam.* Soc. de Teinture et d'Apprêt ci-dev. A. Clavel, and F. Lindemeyer. Fr. Pat. 480,736, Jan. 22, 1916.

SEE Eng. Pat. 101,855 of 1916; this J., 1916, 1215.

*Process of producing nitrocellulose.* Eng. Pat. 9547. See XXII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Dyeing of wood; Theory and practice of the—* F. Moll. Z. angew. Chem., 1916, 29, 405–406.

OWING to the cells in wood being kept together by a layer of hemicelluloses, the dyeing of wood is not the same as the dyeing of cotton cellulose, in which the cells are completely separated; the process also differs from the impregnation of wood with preserving agents, which must be soluble to be effective as antiseptics, whereas the aim in dyeing is to convert the dyestuffs into an insoluble form. By immersing wood in a solution of the dyestuff in a solvent, such as water, alcohol, turpentine, benzene, or acetone, and allowing the solvent to evaporate, the colour penetrates to a depth of 0.5 to 2 mm. This process is not satisfactory for oak, but by continued immersion in aqueous solutions pinewood is penetrated to a depth of 5 to 20 mm. in 8 days; the penetration may be accelerated by using warm solutions, and the wood may be steamed before dyeing. Maple, lime, and poplar give good level light shades; pear tree wood is used for making imitation ebony; oak is shaded to resemble "old oak," or green; walnut, mahogany, and hirc are "aged" by such a process. For technical purposes red beech is generally used on account of the readiness with which it is penetrated. A veneer only requires quite a small pressure; beech and pine require about 5 atm.; oak and similar wood is only penetrated to a depth of 1 mm. even at 100 atm. For complete penetration of pieces of larger cross-section, the filtration (Boucherie) process is used, in which the dye solution is introduced under pressure in the direction of the wood fibre to replace the sap in freshly hewn wood, after steam-

ing until the condensed water is clear. The air in the interior is considered to dissolve when a certain pressure is attained. The structure of wood is considered in relation to the dyeing process. It is preferable to use dyestuffs of a colloidal character that are readily coagulated; the greater their colloidal nature, the less the diffusibility, but the greater the fastness to water and, generally, to light. Additions may be made to the dyebath in order to accelerate diffusion by increasing the colloidal condition of the cell walls; this may also be done by steaming, which does not, however, penetrate sufficiently in the case of hard woods. In order to prevent coagulation, acids are added to solutions of acid dyestuffs and ammonia to those of basic dyestuffs. After treatment in the dyebath, the wood is allowed to stand for 6–12 hrs. to allow the dyestuff to coagulate, and is then allowed to dry or is steamed. Basic dyestuffs of low molecular weight are not fast to light, and coagulate too rapidly on the fibre, so that it is difficult to obtain level shades.—F. W. A.

*Dyeing and cleaning; Studies in*—D. B. Lakc. J. Phys. Chem., 1916, 20, 761–808.

WHEN a dyed material is treated with a solution of another dyestuff, the colour of the first dyestuff may, under suitable conditions, be masked by that of the second dyestuff, but the apparent displacement is due merely to the solvent or peptising action of the water and is independent of the second dyestuff. Probably on account of coagulation of the dyestuff, dyeings made at a high temperature do not bleed so readily as those obtained at a lower temperature. Examples are given of selective adsorption by wool from mixtures of dyestuff solutions at different temperatures. Probably due to the coagulating effect of acid, acid dyestuffs (on wool) bleed less in hot water if dyed from an acid bath. The laws of adsorption of an acid dyestuff in various acid baths as suggested by Bancroft (this J., 1914, 197, 349) hold when the "saturation capacity" of the fibre has been so decreased that the "cutting down" effect of the various anions can be manifested. In the case of the dyestuffs examined, the more the dyestuff is irreversibly adsorbed the less completely and readily is that dyestuff adsorbed at a low temperature, and the less it will bleed, other factors being the same.

The methods used for the removal of stains have been classified as follows: (1) mechanical removal; (2) solution in a liquid; (3) peptising in a liquid, e.g., removing dyes with hot water; (4) peptising with a solution, e.g., removing dyes with sodium carbonate solution; (5) peptising with peptised colloid, e.g., removing stains with soap; (6) peptising in two stages, e.g., removing paint by treatment first with oil and casein and then with soap; (7) adsorption by solid, e.g., in removing grease with fullers' earth or blotting paper, which is considered of special interest; (8) peptising with a liquid and adsorption by solid; and (9) change of substance forming the stain. A large number of examples of practical importance are tabulated, e.g., the removal of soot from cotton by dilute caustic soda is explained as due to the peptisation of the soot by the preferentially adsorbed hydroxyl ions forming a caustic soda-soluble colloid of soot.—F. W. A.

#### PATENTS.

*Cotton fabrics; Production of figured effects on*—Heberlein et Cie. First Addition, dated Nov. 30, 1915, to Fr. Pat. 468,642, Feb. 19, 1914 (this J., 1914, 900). Under Int. Conv., July 6, 1915.

THE cotton fabric, previously mercerised, is submitted in places to the action of sulphuric acid above 50–6° B. (1.54 sp. gr.) in strength, then washed and again mercerised.—B. N.

*Weighting, fixing, mordanting, dyeing and bleaching natural silk, artificial silk, cotton, linen, and other textile materials (by means of foam); Process for*—Soc. de Teinture et d'Apprêt ci-dev. A. Clavel, and F. Lindenmeyer. Fr. Pat. 480,764, Jan. 25, 1916.

FOR the production of foam by means of acid, neutral, or alkaline liquids, saponins, agglutinants, tannins, resins or other analogous substances are added to the liquids.—B. N.

*Silk; Protective treatment of weighted—in order to increase its strength and elasticity.* Schadd & Korteling. Fr. Pat. 480,206, Nov. 12, 1915. Under Int. Conv., Nov. 23, 1914. (See also Fr. Pat. 478,007, Mar. 12, 1915; this J., 1916, 1107.)

SILK, after weighting with tin phosphosilicate, for instance, is treated with organic compounds containing nitrogen or sulphur, or both, which are non-volatile and which are oxidised more readily than the fibroin but do not stain nor injure the feel and appearance of the silk; the compounds employed may be fixed in the fibres by precipitation in an insoluble form. Suitable substances are choline, betaine, and the alkaloids; for example, the silk after weighting is treated with a solution containing 5% of choline and hydrazine hydrochloride.—J. F. B.

*Finishing materials dyed with vat colours; Process of*—A. Kertesz, Mainkur, Germany, and E. Kur, Manchester. U.S. Pat. 1,207,414, Dec. 5, 1916. Date of appl., Mar. 18, 1915.

THE dyed fabric is treated with a finishing paste containing dextrin and an oxidising salt.—F. W. A.

*Mercerised fabrics; Apparatus for leaching and rinsing—and recovering the lye.* J. Matter. Laaken, Assignor to J. P. Bemberg A.-G., Barmen-Rittershausen, Germany. U.S. Pat. 1,209,465, Dec. 13, 1916. Date of appl., Jan. 28, 1913.

SEE Eng. Pat. 1633 of 1913; this J., 1913, 908.

*Dyeing and like apparatus; Perforated beam for*—J., T., and E. Brandwood, Bury. U.S. Pat. 1,211,068, Jan. 2, 1917. Date of appl., Oct. 27, 1914.

SEE Eng. Pat. 17,355 of 1914; this J., 1915, 26.

*Foam for the treatment of textiles; Production of—at any desired temperature. Process for treating textiles, at any desired temperature, with oxidising, reducing, or inert gases in the form of foam.* Soc. de Teinture et d'Apprêt ci-dev. A. Clavel and F. Lindenmeyer. Fr. Pats. 480,397 and 480,398, Dec. 6, 1915.

SEE Eng. Pat. 102,310 of 1916; this J., 1917, 80.

*Process for utilising osier-bark.* Fr. Pat. 480,637. See V.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid from copper-smelting gases.* E. L. Larson. Eng. and Min. J., 1916, 102, 1121–1125.

FIVE important American copper smelting works manufacture acid from furnace gases. The Mountain Copper Co. (Mococo, Calif.), the Anaconda Copper Co. (Anaconda, Mont.), and the Garfield Copper Co. (Utah) use gas from McDougall furnaces roasting sulphide fines. The Tennessee Copper Co. (Copperhill) and the Ducktown Sulphur, Copper, and Iron Co. (Isabella, Tenn.) use gas from blast furnaces in which pyritic smelting is carried out with addition of 4–6% of coke on undressed ores carrying about 30% of pyrrhotite, 6 of pyrite,

and 8 of chalcopryite. The normal composition of the gas from the blast furnaces is  $\text{SO}_2$  8-9,  $\text{CO}$  6-7,  $\text{CO}_2$  0-8-1, and  $\text{N}$  84%. Air is admitted at several points in the acid plant by low-pressure blowers to furnish the necessary excess of oxygen without unduly diluting the sulphur dioxide. Much skill and attention is required at the furnaces, especially in charging, to keep the quality and quantity of the gas as uniform as possible. The nitre consumption in the two plants is 6-8%. All the smelters use the chamber process because it is cheaper and the gas too impure for the contact process; the use of the latter would involve the removal of moisture, arsenic, and dust.—W. R. S.

*Phosphoric acid evolved in the volatilisation method of treating phosphate rock; Use of the Cottrell precipitator in recovering*—W. H. Ross, J. N. Carothers, and A. R. Merz. *J. Ind. Eng. Chem.*, 1917, 9, 26-31.

In the Cottrell precipitator (this *J.*, 1911, 1037) used in the authors' experiments on a manufacturing scale (this *J.*, 1916, 1154) the sheet iron pipes were replaced by glazed terra-cotta tile pipes, within which were suspended wires of Monel metal or nichrome wires weighted at the ends. The exhaust-fan for drawing off the fumes was made of antimonial metal, the surface of which was protected from the action of the phosphoric acid by a coating of Bakelite enamel. Air was admitted to the furnace through openings round the electrodes, whilst the charge was introduced through openings round other electrodes. The rock used in the experiments was unground Florida land-peat phosphate, containing 32.14% of phosphorus pentoxide and 2.33% of fluorine. The amount of moisture in the charge and in the admitted air was sufficient to convert the phosphorus pentoxide into phosphoric acid, and by increasing or reducing the moisture in the air, acid of any degree of dilution could be obtained. When the fumes were passed through a baffle tower so that the precipitation was made at a low temperature, the acid first collected from a charge containing about 1% of moisture had a concentration of about 60%, but by reducing the flow of air the concentration was increased to about 80%. By omitting the baffle tower and passing the fumes directly into the precipitator, precipitation of the phosphoric acid took place at about 100° C., and the solutions obtained either with a maximum or minimum flow of air were more concentrated than those obtained under corresponding conditions at a lower temperature (*e.g.*, 90-82 and 95-23% as compared with 63-07 and 80-23% respectively). The extreme limits of impurities found in samples of phosphoric acid (about 63%) collected in the precipitator were as follows:—Sulphuric acid, 0-04 to 0-21; hydrofluoric acid, trace to 0-49; hydrochloric acid, nil to 0-04; silica, 0-03 to 0-26; calcium oxide, nil to 0-03; ferric oxide and alumina, trace to 0-06; arsenic, trace; heavy metals, none; and residue on ignition, 0-11 to 0-85%. Under the conditions of the precipitation the greater part of the hydrofluoric and sulphuric acids present passed through the precipitator in the form of gas. The phosphoric acid collected when the baffle tower was omitted contained a larger amount of suspended matter and was somewhat coloured probably by carbonaceous matter carried over with the air.

—C. A. M.

*Hydrogen bromide; The absolute density of gaseous*—C. K. Reiman. *Comptes rend.*, 1917, 164, 44-46.

DETERMINATIONS of the density of gaseous hydrogen bromide prepared by three different methods gave as the result of a large number of experiments a mean value of 3.6442 grms.  $\pm 0.002$  as the weight of a litre under normal conditions.—G. F. M.

*Salt; Removal of barium [chloride] from brines used in the manufacture of*—W. W. Skinner and W. F. Baughman. *J. Ind. Eng. Chem.*, 1917, 9, 18-26.

SAMPLES of the brine from different wells in the Ohio River District contained from 61.73 to 85.13 grms. of sodium chloride, 0.23 to 0.60 grm. of barium chloride, and 0.04 to 0.11 grm. of ferrous bicarbonate per litre. The brine, which has a density of 8.5 to 10° B., is concentrated in an evaporator to about 16° B., and is then passed through filters into settling tanks containing copper steam pipes, whence, after concentration to about 20° to 21° B., it is run into a "draw settler." Here a little salt is allowed to crystallise to remove the last traces of iron in suspension, and the clear brine is then drawn off to the crystallising tanks, or "grainers," which contain a system of copper steam pipes. As the salt crystallises more brine is run in until the concentration reaches about 30° B. From these grainers, in which No. 1 or "Table and Dairy Salt" is made, the brine is drawn off into the "bitter water grainer," where it is concentrated to 38° or 40° B. and yields No. 2 or "off-grade" salt, whilst the final mother liquors are drawn off and treated for the recovery of bromine, and calcium and magnesium chlorides. Samples of No. 1 salt contained from 0.02 to 0.23% of barium chloride (calculated on the anhydrous salt), whilst "off grade" salt contained from 1.02 to 18.52%. The "off-grade" product constitutes from 7 to 12% of the total salt produced by a works, and is sold for the manufacture of ice, salting of hides, etc., but notwithstanding its dangerous character, it has not infrequently been substituted for the No. 1 salt. Experiments on the precipitation of the barium by means of sodium sulphate showed that by the addition of the equivalent quantity of the reagent only 81.9% of the total barium was precipitated from the brine, whilst by the addition of 30% in excess 93.7% of barium was removed. The presence of the ferrous bicarbonate, the iron in which was partially precipitated on aeration or boiling, promoted the subsidence of the barium sulphate, and for this reason the sodium sulphate solution was added to the brine before heating. In practice the sodium sulphate (salt-free from arsenic or lead) was dissolved in water and its acidity neutralised by the addition of slaked lime which had been rubbed to a paste. The calculated quantity of solution was added to the brine, which was then agitated by the admission of compressed air for an hour, until the ferrous bicarbonate was decomposed. After standing for 16 hours practically the whole of the barium sulphate had deposited, and the brine was ready for the evaporator. After 3½ months' work scale due to calcium sulphate began to form on the pipes in the grainers, and it was therefore decided to reduce the proportion of sodium sulphate to the amount equivalent to the barium. Under these conditions No. 1 and No. 2 salts were whiter than before owing to more complete removal of the iron, and they crystallised more rapidly in the grainers. The highest proportion of barium found in No. 2 salt was 0.06%, whilst a sample of the mixed No. 1 and No. 2 salts contained only 0.02% of barium sulphate. The cost of treatment is estimated at about 1 cent per barrel, and the increased value of the product more than compensates for this. In the works which adopted this treatment about 400 lb. of barium sulphate per day was obtained as a by-product.—C. A. M.

*Ammonium salts; Exportation of*—

AN Order in Council, dated January 19th, orders that the Proclamation of May 10th, 1916, prohibiting the exportation of certain articles from

the United Kingdom, be altered as follows:—The heading "Ammonia and its salts, whether simple or compound (except ammonium nitrate, perchlorate, and sulphocyanide)," is deleted and replaced by the headings "Ammonia and its salts, whether simple or compound (except ammonium nitrate, perchlorate, sulphate, and sulphocyanide)." "Sulphate of ammonia."

(Note: The effect of this order is to permit the exportation of ammonium sulphate to France and its possessions, Russia, Italy and its possessions, Spain, and Portugal, and to any other destination not in Europe nor on the Mediterranean and Black seas.)

*Ammonium nitrate solutions of any desired strength; Tables for the preparation of* — C. Gautsch. (Chem.-Zeit., 1916, 40, 1049—1050.

TABLES are given showing the quantities, by weight and by volume, of ammonia solution of concentration from 9.91 to 30.37%  $\text{NH}_3$  (sp.gr. 1.060—0.894 at 15° C., 16°—27° B. at 12.5° C.) and of nitric acid of concentration from 10.63 to 68.90%  $\text{HNO}_3$  (sp.gr. 1.060—1.514 at 15° C., 31.7—48.09° B. at 15° C.) to furnish 1 grm. of ammonium nitrate. The following figures have been selected from the tables:—

#### Ammonia solution.

Sp. gr., 15° C. ....	0.960	0.950	0.940	0.930	0.920	0.910	0.900	0.894
Amount required for 1 grm. ammonium nitrate { grms.	2.1489	1.6716	1.3625	1.1425	0.9791	0.8522	0.7517	0.7012
{ c.c.	2.2385	1.7596	1.4495	1.2285	1.0642	0.9365	0.8352	0.7843

#### Nitric acid solution.

Sp. gr., 15° C. ....	1.060	1.120	1.180	1.240	1.300	1.360	1.430	1.480	1.514
Required for 1 grm. ammonium nitrate { grms.	7.3693	3.8905	2.6789	2.0535	1.6373	1.3671	1.1276	0.9146	0.7858
{ c.c.	6.9522	3.4736	2.2702	1.6577	1.2749	1.0052	0.7941	0.6180	0.5256

*Seaweeds [Japanese —].* K. Miyama. Kōgyō-Kwagaku-Zasshi, 1916, 19, 1044—1065.

IN Yezo different species of *Laminaria* and *Phlocladus* are collected for use as edibles and in large quantities as raw material for iodine manufacture; potassium salts are also obtained as a by-product. The cost of potassium chloride from seaweed is less than £4 per ton. Some of these seaweeds are rich in iodine, e.g., *A. bifidus*, Rupr. contains 0.6% on the dry weight. Near the coast of Saghalin seaweeds are as abundant as in Yezo and equally rich in iodine. Other varieties commonly used as a source of iodine are *Ecklonia* and *E. bicyclis*. Kelp made from seaweed in Japan is not porous, but obviously over-roasted. According to experiments, the seaweed may lose 2.5% of its iodine-content by unsuitable roasting. Japan produces 1700 tons of potassium salts per annum, or about one-fourth of the total consumption, but the proper utilisation of the seaweed gathered should afford over 6000 tons.

—J. F. B.

*Cyanides [of the alkali and alkaline-earth metals]; Synthesis of — in the electric pressure-furnace.* IV. Reactions at high pressures. A. Stähler. Ber., 1916, 49, 2292—2294.

MIXTURES of alkali carbonate and carbon, heated in the presence of nitrogen, at 60 atmos. pressure, in the author's high-pressure electric furnace (this J., 1913, 789), furnished a product containing at least 74% of alkali cyanide (yield 95%), and lithium cyanide was obtained by the same method in 80% yield, whilst a product containing about 71% of barium cyanide, with no appreciable proportion of cyanamide, was similarly prepared from barium oxide, the yield of cyanide being over 80%.

Attempts to prepare magnesium cyanide and glucinum (beryllium) cyanide were unsuccessful; and, as regards yield, the synthesis of aluminium nitride was unaffected by working under high pressure, no cyanide being formed.—F. SODN.

*Ammonium silicate.* R. Schwarz. Ber., 1916, 49, 2358—2364.

HYDRATED silica was slowly dissolved by 3 N ammonia solution at 18° C. until a maximum concentration representing about 1/50th of that required for the conversion of all the ammonia into metasilicate was reached. On heating, the silica dissolved more rapidly, but the solutions obtained were opalescent, and the solubility varied with the degree of hydration and character of the silica used. The formation of ammonium silicate was however proved by the steady rise in electrical conductivity which accompanied solution. Ammonia solution had no appreciable effect on finely powdered quartz at the ordinary temperature, but anhydrous amorphous silica underwent slow hydration and therefore dissolved. Tetraethylammonium metasilicate was obtained as a white amorphous hygroscopic mass by dissolving hydrated silica at 80° C. in a 10% solution of tetraethylammonium hydroxide and evaporating the solution in a vacuum desiccator.—F. SODN.

*Iron ammonium alum; Hydrolysis of —.* W. N. Rac. Chem. Soc. Trans., 1916, 109, 1331—1339.

THE precipitate, formed by hydrolysis, which separates on keeping a solution of iron ammonium alum, has been shown to have the composition  $2\text{Fe}_2\text{O}_3 \cdot \text{SO}_3$ . The intensity of the colour of iron ammonium alum solutions, alone and in the presence of other substances, such as sulphuric acid, ammonium sulphate, sucrose, potassium chloride, and alcohol, has been measured, and is shown to be in agreement with the supposition that the colour is due to the presence of a soluble form of the above basic sulphate. On adding concentrated sulphuric acid to a solution of the alum, a white precipitate is formed which is found to be the anhydrous alum. Several density determinations of aqueous iron ammonium alum solutions are recorded.—B. N.

*Compounds formed by the interaction of glacial acetic acid and ferric chloride; Constitution of —.* R. Weinland and K. Kessler. Tübinger Chem. Ges., June, 1916. Chem.-Zeit., 1916, 40, 1002.

ON dissolving ferric chloride in a small amount of glacial acetic acid the compound  $\text{Fe}(\text{CH}_3\text{COO})_3 \cdot \text{Cl} + 2.25\text{H}_2\text{O}$  is formed, whereas when much acetic acid is present the product has the composition,  $\text{Fe}(\text{CH}_3\text{COO})_3 + 0.25\text{CH}_3\text{COOH} + 0.75\text{H}_2\text{O}$ . In both compounds the complex hexa-acetato-dihydroxyferri cation is present (Ber., 1902, 42, 3381) since the concentrated aqueous solutions yield the characteristic platinumochloride of that cation. Treated with alcohol both salts yield a new red rhombohedral compound,  $[\text{Fe}_2(\text{CH}_3\text{COO})_6(\text{OH})_2] (\text{FeCl})_2 + 5 \text{C}_2\text{H}_5\text{OH} + \text{H}_2\text{O}$ , whilst when crystallised from a little water both

form the chloride of the hexa-acetato-triferric base  $[\text{Fe}_3(\text{CH}_3\text{COO})_6(\text{OH})_3]\text{Cl} \cdot 7\text{H}_2\text{O}$ . An analogous compound also separates from a concentrated aqueous solution of 1 mol. of ferric chloride and 1 mol. of sodium acetate, while a solution of 1 mol. of ferric chloride and 2 mols. of sodium acetate yields the above-mentioned chloride of the hexa-acetato-triferric base.—C. A. M.

*Refractory oxides.* R. B. Sosman. J. Ind. and Eng. Chem. 1916, 8, 985—990.

THE author quotes the melting points of silica, alumina, magnesia, lime, and ferrosilicic oxide previously determined by C. W. Kanolt (this J., 1913, 1155) and reproduces the phase rule diagrams of all possible two and three-component systems made from the foregoing oxides and ferric and ferrous oxides. He concludes that the stable compounds of the oxides with each other are all made up of these oxides in simple proportions, usually 1:1 or 2:1 and that the stable compounds are molecular compounds of the oxides (Werner's compounds of the second and third order). He suggests that certain compounds which exist in the crystalline state may be entirely dissociated when converted into the liquid or glassy state. The maximum melting points in all of the systems are those of the pure stable compounds, and the highest melting points are those of the pure oxides,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ , and  $\text{MgO}$ , so that the addition of any substance to a refractory oxide will lower its melting point. Kaolinite  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ , is not the hydrate of any high temperature compound and when heated it breaks up into a mixture of  $\text{Al}_2\text{SiO}_5$  and  $\text{SiO}_2$  with a softening point below that of either constituent, i.e., below  $1625^\circ\text{C}$ . If alumina is added to a pure kaolin to make a mixture equivalent to  $\text{Al}_2\text{SiO}_5$ , sillimanite will form and will not melt below  $1815^\circ\text{C}$ . The formation of this compound is the basis of the manufacture of Marquardt porcelain. Magnesia spinel,  $\text{MgO} \cdot \text{Al}_2\text{O}_3$ , is a similar product; it melts at  $2135^\circ\text{C}$ , and its eutectic with  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  melt respectively at  $2030^\circ\text{C}$ . and  $1925^\circ\text{C}$ . Fused silica or "quartz glass" is only stable above the melting point of cristobalite ( $1625^\circ\text{C}$ ). Below this temperature it tends to crystallise (devitrify), but as the high temperature form of cristobalite has the same density as the glassy form, the material remains transparent and homogeneous and only breaks up into the familiar devitrified mass when passing the  $\alpha$ - $\beta$  inversion point at  $200^\circ$ — $275^\circ\text{C}$ . Future progress in the use of refractory oxides must follow two principal lines: (1) control of the purity of the materials and (2) accurate control of high temperatures.

—A. B. S.

*Hydrogen-oxygen mixtures; Catalysis of — at the ordinary temperature by moist contact substances.* K. A. Hofmann and R. Ebert. Ber., 1916, 49, 2369—2389.

THE catalysis of a gaseous mixture of oxygen and hydrogen brought about by the action of contact substances moistened with aqueous solutions (see Hofmann and Schneider, this J., 1916, 45) is shown to be an electrochemical process, similar in character to that taking place in a Groves' gas battery. It is supposed that parts of the contact surface (of palladised or platinised earthenware) are converted into oxygen electrodes and parts into hydrogen electrodes by contact with the gases. With surfaces of platinum, iridium, or palladium, the activity of the oxygen develops very slowly as compared with that of the hydrogen, and hence, since the rate of catalysis depends on the product of these activities (that is, on the E.M.F. of the system), the maximum effect is only reached by employing more than one contact substance, so as to secure rapid activation both

of oxygen and hydrogen. The best results were obtained by using a palladium film (on earthenware) in conjunction with small quantities of finely divided palladium (reduced by hydrogen from palladous chloride solution) in presence of sodium bicarbonate solution. With such combined catalysts, the extent of the action is far greater than with both catalysts working singly. Under the most favourable conditions, above  $70\text{ c.c.}$  of gas was catalysed in 10 minutes with  $15\text{ sq. cm.}$  of contact surface. A palladium film alone gave the highest figures in an acid solution, which raises the oxidation potential of the oxygen, and the effectiveness of the catalyst was increased by charging it with oxygen, but no rise in activity was induced by adding an oxygen-carrier.

—F. SODN.

*Metallographic investigation of the system, antimony sulphide and cuprous sulphide.* Chikashige and Yamauchi. See X.

*Ionisation of sulphuric acid and normal potassium sulphate in aqueous solutions of medium concentration.* Muller. See XI.

*Remarks on certain electrolyses.* Muller. See XI.

*Use of diphenylamine and diphenylbenzidine for colorimetric determinations.* Smith. See XXIII.

#### PATENTS.

*Hydrochloric acid; Process for the manufacture of pure —.* M. Lucron and C. E. Riche. Fr. Pat. 480,247, Nov. 16, 1915.

THE impurities of ordinary commercial hydrochloric acid, consisting of iron, arsenic, antimony, sulphurous acid, and anhydride, can be removed by treatment of the acid with persalts, notably permanganates. The impurities are thus converted either into insoluble compounds, or into non-volatile compounds not carried away under the conditions of operation. The process can be applied in the ordinary course of manufacture so as to yield, directly, a pure acid.—J. B. C. K.

*Sulphuric acid; Manufacture of —.* L. Laufer. Fr. Pat. 481,131, Mar. 7, 1916.

THE gases escaping from the Gay-Lussac towers are treated with water sprays or the like to absorb acid fumes, etc., and the water containing the recovered products is introduced into the acid-making system.—T. H. B.

*Ammonia from coke; Process for production of —.* J. G. Aarts, Dongen, Holland. Eng. Pat. 101,154, Aug. 8, 1916. Under Int. Conv. Aug. 9, 1915. (Appl. No. 11,202 of 1916.)

A HOT mass of coke, after its discharge from the carbonising chamber, is exposed over its whole length and at both sides, to jets or streams of steam, preferably superheated, directed towards the middle of the mass, under constant conditions of quantity, velocity, and temperature, both for the steam and coke. The ammonia is recovered in any known manner by the aid of hoods, connected if desired with suction apparatus. The velocity and quantity of the steam should be regulated in accordance with the velocity and temperature of the coke, so that a sufficient quantity of nascent hydrogen is formed to give the maximum yield of ammonia, and that the temperature of the coke is reduced sufficiently to prevent as far as possible decomposition of the ammonia formed. In many cases, especially when the coke is moving slowly, the steam is introduced intermittently.—J. B. C. K.



**Sodium bicarbonate and ammonium nitrate; Process of manufacture of —.** F. Ricard. Fr. Pat. 480,082, Oct. 20, 1915.

The process is based upon the reaction between sodium bicarbonate and sodium nitrate, and the sodium bicarbonate and ammonium nitrate. One-third of the sodium nitrate is left undecomposed in the mother liquor, together with ammonium nitrate. The patent covers several alternative methods and apparatus for carrying out the reactions, and for separating the salts from the mother liquor. The ammonium bicarbonate and sodium nitrate may be allowed to react under pressure at 50° C. in the presence of only a small amount of water, or a saturated solution of the salt may be allowed to act under the same conditions as regards temperature and pressure, on the other salt in the dry state. The dried mixture obtained from the mother liquor is extracted with hot water, which dissolves ammonium nitrate in preference to sodium nitrate, and the dried salt obtained by recrystallisation of this solution, is extracted with cold water, to remove sodium nitrate. The residual ammonium nitrate is separated and dried by filtering under pressure of hot air. In order to facilitate the extraction of the raw or refined mother liquor by cold water, the latter is passed repeatedly through the salt by means of a pump, a jet of compressed air, or some mechanical form of agitator. —J. B. C. K.

**Ammonium nitrate; Manufacture of —.** P. L. Hulien. Fr. Pat. 480,150, Feb. 11, 1915.

An aqueous solution of calcium nitrate obtained, e.g., by the electric arc process of fixing atmospheric nitrogen, is allowed to react with ammonium sulphate at a temperature of 120° C. or higher. The solutions are preferably raised to this temperature separately, and the preliminary heating and the reaction are effected under pressure in a series of autoclaves. The apparatus may be applied to other reactions at high temperatures under pressure, notably those producing a precipitate of calcium sulphate. —J. B. C. K.

**Ammonium chloride; Direct process of manufacture of — from chlorine, nitrogen, and hydrogen.** C. L. Mayer. Fr. Pat. 480,232, Nov. 15, 1915.

A mixture of hydrogen, nitrogen, and chlorine gases in definite proportions by volume, is submitted to the action of the silent electric discharge. In order to facilitate the reaction and to remove the products before decomposition can occur owing to its reversal, water or steam is injected under pressure into the chamber in which the reaction occurs. The following proportions are given as suitable for the process: —nitrogen 1 vol., hydrogen 4 vols., chlorine 1 vol. —J. B. C. K.

**Sodium perborate; [Electrolytic] manufacture of —.** Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler, Frankfurt, Germany. Eng. Pats. (A) 100,154, Mar. 2, 1916. Under Int. Conv., Mar. 10, 1915. (Appl. No. 3184 of 1916.) (B) 101,820, Mar. 2, 1916. Under Int. Conv., Sept. 24, 1915. (Appl. No. 3186 of 1916.) Additions to Eng. Pat. 100,153 of 1916 (this J., 1917, 83.)

(A) In the preparation of sodium perborate by electrolysis of a solution of alkali carbonate and alkali borate, the solution, which may be saturated or not with borax, is kept saturated with alkali carbonate by maintaining the latter as a "bottom substance" (solid phase). (B) During electrolysis under the above conditions, a substance or mixture is added which will produce on the cathode a precipitate lowering the reducing action. Chromic acid may be used for this

purpose, and such of its salts the base of which does not decompose alkali perborate, e.g., chromates of alkali and alkaline earth metals, including magnesium, aluminium, beryllium (glucinum), etc. Other alkaline earth salts may also be used. With chromic acid 0.5 grm. per litre, and with calcium chloride 1 grm. per litre may be used. Protective colloids, e.g., albumin, starch, gum arabic, &c., may also be introduced to increase the action of the added chromic acid or salts. —T. H. B.

**Iodine; Direct method of extraction of — from green sea-weed.** V. Vincent. Fr. Pat. 480,014, Oct. 16, 1915.

GREEN sea-weed is extracted in the undried state with a solution of aluminium sulphate, preferably in sea water, and slightly acidified, which has the property of dissolving the alkali iodide from the weed, while leaving undissolved the gelatinous organic matters. The solution of alkali iodide is treated with copper sulphate and with sulphur dioxide or sulphites, and the precipitated cuprous iodide is separated from the solution by centrifugal force. An alternative method of extraction is to use a solution of iron sulphate for lixiviating the dried sea-weed. —J. B. C. K.

**Sulphur dioxide; Synthetic manufacture of —.** R. P. Pictet. Fr. Pat. 480,294, Nov. 22, 1915.

SULPHUR or pyrites is burnt in a special furnace by means of pure oxygen, and to prevent the formation of products other than sulphur dioxide, the oxygen is mixed with sulphur dioxide before coming in contact with sulphur, and cooling coils are embedded in the walls of the furnace to moderate the temperature. A baffle-plate is fixed longitudinally in the furnace to divert the gaseous stream in a suitable manner. A compressor and exhauster are used to work an injector for mixing the oxygen with the sulphur dioxide produced in the process before bringing it in contact with the sulphur. Sulphur is kept in slight excess during the process so that all the oxygen is consumed. —T. H. B.

**Zinc sulphate; Production of — from zinciferous minerals.** E. Ferraris. Fr. Pat. 480,409, Dec. 21, 1915. Under Int. Conv., Dec. 7, 1915.

THE roasted and desulphurised ore or natural zinc oxide is treated with sulphur dioxide produced, e.g., by the roasting of a sulphide ore, and the precipitated zinc sulphite is treated with moist air to convert it into sulphate, which is dissolved by lixiviation with pure or acidulated water. The two stages of the process may be combined in one operation, treating the roasted ore with sulphur dioxide, together with air and moisture (or steam) in such a way as to produce directly zinc sulphate, the residue being afterwards extracted with acidulated water. —T. H. B.

**"Mixed cream of tartar" [potassium and sodium bitartrates] and "soda cream of tartar" [sodium bitartrate]; Industrial preparation of —.** T. Gladysz. Fr. Pat. 480,620, Mar. 12, 1915.

CRUDE tartar is treated with hydrochloric acid, the acid solution is partially neutralised with lime or calcium carbonate, the precipitate of potassium and calcium bitartrates is separated and boiled with sodium carbonate solution, the resulting solution is decolorised with animal charcoal, and treated with a mineral acid to precipitate a mixture of sodium and potassium bitartrates ("mixed cream of tartar"). By exact neutralisation of the first mother-liquors with neutralisation of the first mother-liquors of the precipitated calcium tartrate by sodium carbonate, and treatment of the lye as described above, a crystalline precipitate of sodium bitartrate ("soda cream of tartar") is obtained. —T. H. B.



**Carbon dioxide; Process for absorption of — from gaseous mixtures.** Naaml. Venoot-schaap Anton Jurgens' Vereenigde Fabrieken. Fr. Pat. 480,774, Jan. 26, 1916. Under Int. Conv., Nov. 8, 1915.

THE gaseous mixture is led into a solution capable of absorbing carbon dioxide, and which can be afterwards regenerated by boiling. The solution is one of alkali carbonate in which magnesium hydroxide, or a highly basic carbonate of magnesia or a mixture of these compounds, is held in suspension. This solution is obtained by adding an excess of a solution of an alkali carbonate to one of a magnesium salt, and afterwards boiling for some time.—T. H. B.

**Alumina and hydrochloric acid; Simultaneous production of —.** L. G. Patrouilleau. Fr. Pat. 481,106, Mar. 2, 1916.

SILICO-ALUMINIUM, produced by reduction of silicious bauxite in an electric furnace, is heated to dull redness in a refractory vessel, and chlorine is brought into contact with it. A mixture of the chlorides of aluminium and silicon results, which is led into suitable condensers, the excess of chlorine which accompanies it being led away and used again. Subsequent treatment of the chlorides is based on the facts that silicon chloride decomposes in aqueous solution into silica and hydrochloric acid, whilst the very soluble aluminium chloride decomposes only after total evaporation of its solution and roasting of the residue at a red heat, then becoming converted into alumina and hydrochloric acid.—T. H. B.

**Oxygen compounds of metals or metalloids and carbonaceous substances; Manufacture of an intermediate product for use in processes based on heating together — [e.g., in production of calcium carbide].** Alby United Carbide Factories, Ltd., and K. Hylland. Fr. Pat. 480,020, Oct. 18, 1915. Under Int. Conv., Oct. 31, 1914.

SEE Eng. Pat. 14,486 of 1915; this J., 1916, 963. The process may be applied to other oxygen compounds of metals or metalloids, besides lime, and the product may be used in the manufacture of metals, alloys, nitrates, silicates, cyanides, and cyanamides, as well as of carbides.

**Sulphuric acid; Process and apparatus for the manufacture of —.** L. P. Basset, Paris. Eng. Pat. 100,597, May 31, 1916. Under Int. Conv., May 31, 1915. (Appl. No. 7724 of 1916.)

SEE U.S. Pat. 1,197,331 of 1916; this J., 1916, 1058.

**Carbon dioxide; Process of absorbing — from gaseous mixtures.** Naaml. Venootschap "Ant. Jurgens' Vereenigde Fabrieken," Oss, The Netherlands. Eng. Pat. 102,138, Feb. 16, 1916. Under Int. Conv., Nov. 8, 1915. (Appl. No. 2331 of 1916.)

SEE Fr. Pat. 480,774 of 1916; preceding.

**Ammonium phosphate; Manufacture of acid —.** W. Wollenweber, Bochum, Germany. U.S. Pat. 1,208,877, Dec. 19, 1916. Date of appl., Oct. 19, 1915.

SEE Eng. Pat. 8037 of 1915; this J., 1916, 737.

**Ammonia; Apparatus for condensing —.** L. Block, Marmaroneck, N.Y. U.S. Pat. 1,270,141, Dec. 26, 1916. Date of appl., Dec. 2, 1912.

SEE Fr. Pat. 444,088 of 1912; this J., 1912, 1113.

**Alkaline compounds; Method of producing soluble —.** P. Radmann, Godegard, Sweden. U.S. Pat. 1,209,201, Dec. 19, 1916. Date of appl., May 23, 1914.

SEE Eng. Pat. 12,136 of 1914; this J., 1915, 138.

**Nitrogen compounds; Apparatus for production of —.** C. Krauss, Cologne-Braunsfeld, and P. Stähelin, Knapsack, Germany. U.S. Pat. 1,211,181, Jan. 2, 1917. Date of appl., Mar. 26, 1915.

SEE Fr. Pat. 464,750 of 1913; this J., 1914, 549.

**Zinc sulphate or other zinc compounds; Manufacture of — from zinc ores.** R. B. Llopert. Fr. Pat. 480,848, Feb. 2, 1916. Under Int. Conv., Apr. 2, 1915.

SEE U.S. Pat. 1,142,795 of 1915; this J., 1915, 834.

**Sulphur; Recovering elemental — from sulphur gases.** W. F. Lamoreaux, Isabella, Tenn., U.S.A. Eng. Pat. 16,543, Nov. 23, 1915. Under Int. Conv., Feb. 8, 1915.

SEE U.S. Pat. 1,169,726 of 1916; this J., 1916, 309.

**Sulphur; Recovery of — from sulphurous gases.** W. F. Lamoreaux. Fr. Pat. 480,345, Nov. 23, 1915. Under Int. Conv., Feb. 8, 1915.

SEE U.S. Pat. 1,169,726 of 1916; this J., 1916, 309.

**Oxygen from air; Apparatus for extraction of —.** H. Filippo, P. Schoonenberg, and Naaml. Venootschap Philips' Gloeilampenfabriek. Fr. Pat. 480,915, Feb. 10, 1916. Under Int. Conv., Oct. 16, 1915.

SEE Eng. Pat. 101,860 of 1916; this J., 1917, 32.

**Automatic pulsometer for raising liquids, especially acids.** Fr. Pat. 480,861. See 1.

**Process for producing a water-softener.** U.S. Pat. 1,207,826. See XIX.

## VIII.—GLASS; CERAMICS.

**Bricks: Yellow —.** L. A. Keane. J. Phys. Chem., 1916, 20, 734—760.

IT has been found that the yellow colour of bricks is due to ferric oxide in a very finely divided form (not to compounds of the latter with lime or silica); when the ferric oxide is in a coarse condition the colour of the bricks is red. Alumina appears to be the important peptising agent in bricks, which are yellow when the ratio of free alumina to iron is high; lime acts indirectly by setting free alumina. Anhydrous yellow ferric oxide has never been obtained in the pure state, but it is stabilised by other substances. The buff colour of the product obtained by igniting aluminium hydroxide containing a small percentage of ferrous hydroxide is undoubtedly due to ferric oxide, as also is the yellow colour of slightly impure quicklime. Clay deposited in the presence of organic matter, and therefore likely to contain very finely divided iron oxide, may burn to a buff colour without the lime or alumina content being high. As agglomeration increases with the temperature, yellow bricks might be expected to turn red if heated sufficiently; but the peptising action of alumina also increases with the temperature, and the latter appears to be the predominating factor, since red bricks become paler or even buff when reheated. The changes of colour which result from the heating of yellow bricks are complicated by the dissociation of the contained ferric oxide, and heating in oxygen is suggested as a means of eliminating this factor.—W. E. F. P.

**Committee on Glass and Optical Instruments.** See page 119.

**Refractory oxides.** Sosman. See VII.

**PATENTS.**

*ina clay; Apparatus for drying*—. C. A. Battiscombe and N. O. Walker, London, and G. L. Bates, Playden, Sussex. Eng. Pat. 102,784, May 11, 1916. (Appl. No. 8747 of 1916.)

THE soft clay paste passes through a hopper and forms a film,  $\frac{1}{16}$  in. to  $\frac{1}{8}$  in. thick, on an absorbent dress band, made of canvas, which conveys it through a shallow tunnel heated by steam-pipes and beneath the upper part of the tunnel. After reaching the further end of the tunnel, the clay is scraped by a scraper and falls on to another band, similarly heated, which conveys it again through the tunnel and discharges it in a dry state.—A.B.S.

*factory materials resistant to corrosion; Manufacture of*—. Mineral Products Co. Fr. Pat. 480,491, Dec. 18, 1915. Under Int. Conv., Dec. 21, 1914, and Aug. 9, 1915.

THE Eng. Pat. 17,447 of 1915; this J., 1916, 692. The proportion of finely divided carbon specified from 10 to 30% of the weight of alumina. A material resisting the action of hydrofluoric acid is obtained by heating the products at a high temperature.

*abrasives; Manufacture of—for cleaning and polishing*. Naamlooze Vennootschap West Bomco Cultuur Maatschappij. Fr. Pat. 481,099, Mar. 1, 1916. Under Int. Conv., Mar. 4, 1915. An abrasive, cleaning, or polishing material is made of acid salts of the heavy metals, such as stannic titanate or chromate (ferro-titanic sand or iron ore), which have been freed from various impurities by sifting and then mixed with sand, charcoal, rouge or other abrasive and with a bond such as rubber, oil, grease, cement, or clay. If great strength is required (as in the manufacture of grinding wheels), the mixture may be moulded and then burned.—A. B. S.

*Recuperative kiln*. U.S. Pat. 1,207,209. See I.

**IX.—BUILDING MATERIALS.**

*Theory and practice of the dyeing of wood*. Moll. See VI.

*Sources of rosin in Germany. [Artificial seasoning of wood.]* Besemfelder. See XIII.

**PATENTS.**

*Paving composition, and method of producing same*. E. H. Staber, Calcutta, India. Eng. Pat. 13,753, Sept. 27, 1915.

A PAVEMENT composition consists of grains of a mineral aggregate of different sizes, graded so as to give the maximum density, and not less than 1% of bitumen. The softening temperature of the bitumen is increased by mixing it with an impalpable powder so that the paving material does not soften under the highest sun temperature. Details of the method of grading, with graphs of seven mixtures, are given.—A. B. S.

*Metallic coating on artificial stones and articles of artificial stone mass; Process for the production of a thick or compact*—. H. Welte, Znaim, Austria. Eng. Pat. 17,862, Dec. 21, 1915. Under Int. Conv., Apr. 10, 1915.

THE surface of the unset mass is strewn with a powdered metal, such as lead, tin, zinc, aluminium, or their alloys, preferably composed of globular or angular particles, before being subjected to the final pressure in the mould by which the desired shaping is produced. A soft and a less soft metal powder may be used in succession, or a mixture

of powdered metals or alloys may be employed, with or without a small proportion of graphite in either case.—W. E. F. P.

*Artificial stones; Manufacture of*—. J. Voisembert and F. H. Keudel. Fr. Pat. 480,285, Apr. 7, 1915.

ARTIFICIAL building stone is made by crushing or grinding natural stone, adding 1–4% of a bond such as felspar, clay, ground glass, or quartz, and sufficient water to make a stiff paste. The latter is pressed hydraulically or mechanically into the desired shapes and the blocks produced are burned in an electric or gas-fired kiln. The blocks are laid in a mortar made of the same materials together with cement or lime.—A. B. S.

*Magnesian cement*. E. Ruch. Fr. Pat. 480,338, Nov. 27, 1915.

MAGNESIAN cement is made by adding sufficient dilute hydrochloric acid to calcined magnesite and inert materials such as stone, sawdust, etc., to form a paste, and then stirring the mixture vigorously.—A. B. S.

*Wood; Bleaching exotic*—. P. E. Simon. Fr. Pat. 481,155, June 15, 1915.

ROSE-WOOD, violet-wood (palisander wood), or other exotic wood is soaked in dilute nitric acid, for about a fortnight if cold acid is used or four days if the acid has a temperature of 40° C. It is then washed in a stream of water for a fortnight or more until all traces of nitric acid have been removed.—A. B. S.

*Lumber; Treatment of—to increase its density*. F. Pfeumer, Dresden, and H. Pfeumer, Loschwitz, Germany. Eng. Pat. 100,792, June 24, 1916. Under Int. Conv., June 25, 1915. (Appl. No. 8916 of 1916.)

SEE Ger. Pat. 291,945 of 1915; this J., 1916, 849.

*Cement; Process and furnace for manufacture of—for preparing concrete of great strength*. E. Longan. Fr. Pat. 480,542, Dec. 9, 1915. Under Int. Conv., Dec. 11, 1914.

SEE Ger. Pat. 293,124 of 1914; this J., 1916, 006.

**X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.**

*Gold in copper matte; Assaying*—. R. E. Chase, jun. Eng. and Min. J., 1916, 102, 1139.

THE method is especially adapted for mattes rich in gold. One-quarter assay-ton is divided between two 3 in. scorifiers containing 35 grms. of test lead and 2 grms. of silica, and a second sample of the same weight is divided between a second pair of scorifiers, the assay being run in duplicate. The contents of the scorifiers are mixed and each charge is covered with 35 grms. of test lead and 2 grms. of borax glass. The lead buttons are combined two and two, made up to 65 grms. with test lead, and re-scorified with 2 grms. of silica. The silver beads are combined two and two and the assay finished in the usual way.—W. R. S.

*Mineral and metal production of the United States*. Eng. and Min. J., Jan. 6, 1917.

THE subjoined tables show the estimated amounts of certain important metals and minerals produced in the United States in 1916, together with the figures for 1914 and 1915:—

## Metals.

Metal.	Unit.	1914.	1915.	1916.
Copper (a) .....	Pounds	1,158,581,876	1,423,608,160	1,941,900,566
Permanganese .....	Long tons	185,118	226,957	355,356
Gold (b) .....	Dollars	94,531,800	101,035,700	92,315,363
Iron .....	Long tons	23,147,220	29,916,213	39,484,000
Lead (c) .....	Short tons	538,735	535,922	583,498
Nickel (e) .....	Pounds	30,067,064	44,139,828	61,675,438
Quicksilver .....	Flasks	16,300	(f) 21,033	28,842
Silver (b) .....	Troy ounces	72,465,100	67,485,600	72,883,748
Zinc (d) .....	Short tons	362,361	492,495	672,306

(a) Production from ore originating in the United States. (b) The statistics for 1914 and 1915 are the final and those for 1916 the preliminary statistics reported jointly by the directors of the Mint and the U.S. Geological Survey. (c) Production of refined metal from ore and scrap originating in the United States; Antimonial lead is included. (d) Total production of smelters, except those treating gross and junk exclusively; includes spelter derived from imported ore. (e) Imports; for 1914, 1915, and 1916 first 10 months only. This nickel is refined in the United States for the production of metal, oxide and salts. (f) As reported by U.S. Geological Survey.

## Mineral and chemical substances.

Substance.	Unit.	1914.	1915.	1916.
Coal, anthracite (a) .....	Short tons	90,821,507	88,612,000	88,500,000
Coal, bituminous (a) .....	Short tons	422,703,370	432,500,000	509,000,000
Coke (a) .....	Short tons	24,555,914	41,600,000	54,300,000
Iron ore .....	Long tons	42,911,897	58,843,804	81,095,000
Petroleum (a) .....	Barrels	265,762,535	281,104,104	292,300,000

(a) The coal and coke and petroleum statistics are the estimates of the U.S. Geological Survey.

**Brass; The failure of —.** I. *Microstructure and initial stress in wrought brasses of the type Cu 80, Zn 40%.* P. D. Merica and R. W. Woodward. U.S. Bureau of Standards, Technical Paper No. 82. J. Franklin Inst., 1916, 182, 803—805.

As the result of examining some 250 samples of used and unused wrought brass of the above type, the initial stresses in rods and bolts were found to vary greatly in distribution and magnitude. Average stresses of 1000—30,000 and fibre stresses of 0—80,000 lb. per sq. in. were obtained (mainly by the Heyn method; see this J., 1914, 596); and whereas the outside layers of extruded and forged rods were found to be under compressional strain, those of drawn rods were in tension. Some of the results obtained for typical materials are given in the following table: samples 85, 136, 172, 174, and 205 were new, 3 and 160 had season-cracked in service, while 187 and 189 had been in service under moderate stress for months without showing signs of failure.

5000—10,000 lb. per sq. in., an average initial stress value of 500 lb. per sq. in. is a safe stress limit for rods and bolts of usual size; and failure is likely to occur in cases where the initial stress value, or the sum of initial stress in tension and the tensional service stress, approaches the elastic limit of the material. The initial stress in rod is relieved by annealing for 1—2 hours at 300°—400° C., at which temperature the physical properties are not appreciably affected.—W. E. F. P.

**Zinc, cadmium, and mercury; Vapour pressure of —.** A. C. Egerton. Phil. Mag., 1917, 33, 33—48.

THE method employed by Knudsen (Ann. Phys., 1909 (4), 29, 179) for determining the vapour pressure of mercury (depending on the kinetic flow of molecules through small apertures and tubes) was adapted to the requirements of the present investigation, the original procedure being simplified by the use of tubes having accurately circular holes the diameters of which

No.	Material.	Percentage composition.				Tensile strength (lb. per sq. in.).		Percentage elongation in 2 ins.	Initial stress (lb. per sq. in.).		Stress in outer layer.
		Cu.	Zn.	Sn.	Fe.	Ultimate strength.	Proportional limit.		Average stress.	Maximum fibre stress.	
3	Manganese bronze	60.0	38.6	0.78	0.5	70,000	17,000	33	25,000	44,000	Tension
85	Naval brass	59.8	39.3	0.61	—	61,000	16,000	46	2,000	7,000	Tension
136	Manganese bronze	59.1	39.3	0.78	0.70	72,000	27,500	44	22,000	34,000	Tension
160	Manganese bronze	57.3	40.7	0.94	1.08	61,000	14,000	28	30,000	84,000	Tension
172	Muntz metal	59.4	40.2	—	0.02	44,000	36,000	(on 3 ins.) 40	4,000	8,000	Compression
174	Manganese bronze	59.9	40.1	1.63	1.18	84,000	36,000	22	4,000	9,000	Compression
187	Naval brass	60.0	39.6	0.40	—	64,600	28,700	33	0,000	14,000	Compression
189	Manganese bronze	56.6	40.8	1.00	1.50	61,600	24,000	—	8,000	12,000	Compression
205	Manganese bronze	58.8	39.6	0.39	1.06	84,600	52,000	22	5,000	9,000	Tension

Failures by fracture or fissure were found to have occurred as the result of (1) the presence of initial stresses of large magnitude, (2) service over-stress due, for example, to the drawing up of bolts too tightly, and (3) improper and faulty practice in forging bolt heads, flanging plates, etc. Under normal service conditions, in which the service stresses are themselves not greater than

could be measured by a micrometer; the condensed metal was weighed instead of measured. The vacuum was obtained by means of a charcoal tube kept in liquid air, and also by the use of a Rose oil pump. The apparatus was standardised by means of mercury; and details of the observations made with all three metals are given in a series of tables. The values of log. p. for cadmium

and zinc were found to be  $10.5979 - 0.5 \log. T$   
 $\frac{6060}{T}$  mm. and  $10.9443 - 0.5 \log. T - \frac{7176}{T}$  mm.,  
 respectively; the corresponding value for mercury,  
 as found by Knudsen, being  $10.5724 - 0.847$   
 $\log. T - \frac{3342.26}{T}$  mm. The angle of slope of the  
 vapour pressure curve for mercury was slightly  
 less than half that of the slopes of the zinc and  
 cadmium curves, which were very similar. The  
 values of  $\frac{dp}{dT}$  at the melting points of the  
 elements were: Zn  $4.41 \times 10^{-3}$ , Cd  $3.70 \times 10^{-3}$ ,  
 and Hg  $2.81 \times 10^{-7}$  mm.; while at a point of  
 equal pressure,  $2.0 \times 10^{-6}$  mm., the corresponding  
 values were  $1.25 \times 10^{-7}$ ,  $1.39 \times 10^{-7}$ , and  $2.81 \times 10^{-7}$   
 mm. at the absolute temperatures  $502^\circ$ ,  $436^\circ$  and  
 $234^\circ$  respectively. At the melting points, the  
 vapour pressures of zinc and cadmium were nearly  
 equal (Zn  $1.13 \times 10^{-1}$ , Cd  $1.0 \times 10^{-1}$  mm.). The  
 physical constants of these two elements are closely  
 similar, but those of mercury have a much smaller  
 value throughout. The results obtained so far  
 show the method to be capable of general applica-  
 tion.—W. E. F. P.

#### Aluminium; Official price of —.

The selling price of aluminium ingots of ordinary  
 commercial purity of 98–99% has been fixed  
 by the Ministry of Munitions at £225 per ton,  
 carriage paid to consumers' works. The maximum  
 selling price of re-melted aluminium scrap and  
 swarf ingots of 98–99% purity has been fixed at  
 £210 per ton, carriage paid to consumers' works.  
 The maximum price must not be exceeded, but  
 a lower one may be fixed by agreement between the  
 seller and the buyer, based upon the metallic  
 aluminium content. These prices are to take  
 effect as from 1st January, and permits under  
 Regulation 30 A of the Defence of the Realm  
 Regulations will be granted only for such dealings  
 in the above-mentioned materials as are in accord-  
 ance with the above prices. The above prices are  
 subject to alteration, at any time, as may be  
 directed by the Minister of Munitions.

#### Gallium; Electrolysis of —. H. S. Uhler. Amer. J. Sci., 1917, 43, 81.

The electrolytic deposition of gallium by the  
 process described recently (this J., 1916, 1221) is  
 considerably retarded by the presence of nitrates.  
 Chlorides, on the other hand, appear to have no  
 effect on the rate of deposition.

#### Antimonial lead; Analysis of —. C. R. McCabe. J. Ind. Eng. Chem., 1917, 9, 42–44.

The following modification of Demorest's method  
 (J. Ind. Eng. Chem., 1913, 5, 842) obviates the  
 error caused by occlusion of antimonious sulphate  
 by the precipitated lead sulphate:—One grm. of  
 the alloy is dissolved in 50 c.c. of strong sulphuric  
 acid, and the solution boiled for 5 mins. to oxidise  
 the tin, and allowed to cool. It is then diluted  
 with 50 c.c. of water, boiled for 10 mins., allowed  
 to cool somewhat, and a further 50 c.c. of water  
 added. The precipitated lead sulphate is washed  
 twice by decantation with 25 c.c. of water, the  
 washings being passed through an asbestos filter  
 in a Gooch crucible, and it is then dissolved in a  
 solution of 10 grms. of ammonium acetate in  
 50 c.c. of water. The solution is treated with  
 25 c.c. of strong sulphuric acid, the flask shaken,  
 and the lead sulphate left to settle for 5 mins. and  
 then collected in the Gooch crucible, in which,  
 after being washed with water, it is dried at  $120^\circ \text{C}$ .,  
 ignited for 5 mins. over a Bunsen flame, cooled in  
 a desiccator, and weighed. The antimony in the  
 two filtrates is separately determined by adding a  
 large excess of  $N/10$  potassium permanganate

solution (standardised on pure antimony dissolved  
 in sulphuric acid) and titrating the excess with  
 standard ferrous sulphate solution. The total  
 amount of permanganate used, less 0.3 c.c., the  
 quantity consumed in a blank test, corresponds  
 with the amount of antimony. Tin is determined  
 in the first filtrate by reduction with iron, filtration  
 of the liquid through cotton wool, which is then  
 washed with dilute (1:5) sulphuric acid, and  
 titration of the filtrate with standard iodine  
 solution.—C. A. M.

#### Burma's wolfram output. U.S. Commerce Re- ports, No. 303, Dec. 27, 1916.

COMPLETED figures for 1915 show that the total  
 production of wolfram from all sources in Burma  
 amounted to 46,293 cwt., compared with 43,752  
 cwt. in 1914, 32,091.5 in 1913, 32,224.5 in 1912,  
 and 25,357 in 1911. These figures do not include  
 production from the southern Shan States, where  
 wolfram is worked in conjunction with tin, and  
 separate statistics are not recorded.

Hitherto the greater portion of the wolfram ore  
 produced in Burma was taken up by Germany, but  
 English smelters are now in a position to handle  
 all the ore coming forward. Late figures show  
 that from January 1 to October 7, 1916, 2702 tons  
 of wolfram was exported, compared with 1844 tons  
 in the corresponding period of 1915. (See also  
 this J., 1916, 119.)

#### Silver-tellurium; Metallographic investigation of the system —. M. Chikashige and I. Saito. Mem. Coll. Sci., Kyoto Imp. Univ., 1916, 1, 361–368.

MIXTURES of silver and tellurium in varying  
 proportions and, in each case, of a total volume  
 of 2.5 c.c., were melted in an atmosphere of  
 hydrogen in a porcelain tube contained in an  
 electric furnace. During the progress of cooling,  
 temperature readings were taken by means of a  
 calibrated thermo-element. An allowance was  
 made for the volatilisation of tellurium, which was  
 found to be appreciable when the proportion  
 present in the mixture exceeded 40%. The forma-  
 tion of homogeneous crystals and eutectoids  
 was followed by maxima and constant temperature  
 points in the cooling curves, and by microscopic  
 examination of the reguli. Formation of the  
 compounds  $\text{Ag}_2\text{Te}$  and  $\text{Ag}_3\text{Te}_2$  was established.  
 $\text{Ag}_2\text{Te}$  is a brittle, greyish-white, crystalline sub-  
 stance melting at  $937^\circ \text{C}$ ., which is immiscible  
 with silver in the solid state and thus forms an  
 eutectoid with this metal, but reacts with tellurium  
 in the molten condition at  $443^\circ \text{C}$ . to form  $\beta$ -  
 $\text{Ag}_2\text{Te}$ , which, at  $403^\circ \text{C}$ ., changes into the  $\alpha$ -form.  
 $\text{Ag}_3\text{Te}_2$  was found to be insoluble in  $\text{Ag}_2\text{Te}$  but  
 remains mechanically mixed with the latter, and  
 immiscible with tellurium in the solid state, so  
 forming an eutectoid when separating together.  
 Reguli containing 70% of silver give an alloy of  
 fine structure which may have technical import-  
 ance.—J. N. P.

#### Metals; Emulsions and suspensions with molten —. H. W. Gillett. U.S. Bureau of Mines. J. Phys. Chem., 1916, 20, 729–733.

FROM a consideration of numerous cases in practice  
 where emulsions or suspensions of molten metals  
 or alloys with solids, liquids, or gases are pro-  
 duced unavoidably, as in the refining of aluminium  
 chips, "floured" or "sickened" mercury, dirty  
 molten sodium, and "blue" zinc powder, or  
 intentionally as in sherardising and in the pro-  
 duction of the lead-copper mixture used for  
 packings, bearings, etc., it is suggested that, if  
 procurable, many stable emulsions of metals  
 normally immiscible in the liquid state might  
 prove of use industrially. Although useful emul-  
 sions of gases with metals are rare, it is known  
 that lead made porous by an indirect method is

much more efficient than solid lead plate for storage batteries; and it is probable that other metals, if rendered uniformly porous by emulsification with gas while in the liquid state, would find many industrial applications. Investigation of the colloid chemistry of molten metals and alloys would doubtless yield much valuable information.—W. E. F. P.

**Antimony sulphide and cuprous sulphide; Metallographic investigation of the system.**—M. Chikashige and Y. Yamauchi. Mem. Coll. Sci., Kyoto Imp. Univ., 1916, 1, 341—347.

AN investigation was made of the crystallisation of mixtures of antimony and cuprous sulphides. Mixtures of varying proportions were melted by means of an electric furnace in tubes of Jena glass or porcelain. Temperatures during the gradual cooling were read by a platinum-platinum-rhodium thermo-element and an atmosphere of nitrogen was maintained round the tube containing 30 grms. of the mixture. Maxima in the cooling curves were obtained with a mixture corresponding to the composition  $4\text{Cu}_2\text{S}, 5\text{Sb}_2\text{S}_3$ , and with  $3\text{Cu}_2\text{S}, \text{Sb}_2\text{S}_3$  (orthothioantimonite). Metallographic examination showed that homogeneous crystallisation took place at these compositions, and the compounds gave no mixed crystals with each other or with either of their components. Indications were obtained of the separation of  $\text{Cu}_2\text{S}, \text{Sb}_2\text{S}_3$  (metathioantimonite), but this formation remained uncertain as the crystallisation was accompanied by the separation of a eutectoid containing  $3\text{Cu}_2\text{S}, \text{Sb}_2\text{S}_3$ , which gave a heterogeneous structure to the regulus. On account of the uncertainty of this crystallisation, the composition of chalcostibnite still remains unsettled. An analysis of the mineral gives 46.81% antimony, while  $\text{Cu}_2\text{S}, \text{Sb}_2\text{S}_3$  contains 48.5% and  $4\text{Cu}_2\text{S}, 5\text{Sb}_2\text{S}_3$ , 45.9%.—J. N. P.

**Sulphuric acid from copper-smelling gases.** Larison. See VII.

**Overvoltage tables. IV. Theories of overvoltage and possibility.** Newbery. See XI.

#### PATENTS.

**Tool-steel alloy.** R. Furness, Jenkintown, and R. H. Patch, Assignors to The Midvale Steel Co., Philadelphia, Pa. U.S. Pats. (A) 1,206,833, (B) 1,206,834 and (C) 1,206,902, Dec. 5, 1916. Dates of appl., Dec. 14, 1915, Jan. 19, 1916, and Sept. 16, 1915.

HIGH speed tool steels are claimed containing (A) Cr, 2.5—5 (4); Mo, 4—11 (8); Co, 2—20 (5); C, less than 1%; (B) Cr, 2—6; W, 9—15 (less than 15); Co, 5—20 (not less than 10%); (C) Cr, 15—20 (19.5); C, about 1.35%.—W. E. F. P.

**Iron for castings; Method of preparing.**—W. G. Kranz, Sharon, Pa., Assignor to The National Malleable Castings Co., Cleveland, Ohio. U.S. Pat. 1,206,861, Dec. 5, 1916. Date of appl., Nov. 17, 1915.

A PORTION of the molten iron, high in carbon, manganese, and silicon, is treated in a Bessemer converter to reduce the amounts of these elements, and the molten iron is then mixed with the remaining portion of the charge, whereby the amounts of the elements are reduced and equalised, though the carbon remains above 1.5%. The metal is refined in an electric furnace, without further substantial reduction of the carbon.—B. N.

**Ferro-tungsten; Process of treating.**—P. M. McKenna, Washington, D.C. U.S. Pat. 1,208,596, Dec. 12, 1916. Date of appl., Aug. 11, 1916.

**FERRO-TUNGSTEN** alloy obtained by smelting is treated for the removal of non-metallic impuri-

ties, such as sulphur, by grinding the mass and subjecting it to the action of hydrochloric acid of approximately 20° B. (sp.gr. 1.16). The acid does not combine with the metallic tungsten alloy, but forms hydrogen compounds, such as hydrogen sulphide, with the impurities. The excess of acid is removed by washing, and the finely divided product dried.—B. N.

**Tin from cassiterite and oxidation products of tin; Process for facilitating the extraction of.**—G. Michaud and E. Delasson, Montreuil sous Bois, France. Eng. Pat. 17,743, Dec. 18, 1915. Addition to Eng. Pat. 614, Jan. 14, 1915 (this J., 1916, 313).

THE reduced metal, obtained as a concentrate or fine powder or in the form of "carbo-metallic plates," by the original process, is dissolved in a solvent composed of hydrochloric acid (40 litres), water (60 litres), and stannous chloride (20 kilos.) at a temperature between 50° and 70° C. The solution is electrolysed for the production of metallic tin, and the spent electrolyte used for dissolving a further quantity of the crude metal. The stannous chloride for the solvent is obtained by treating the partly exhausted carbo-metallic plates with hydrochloric acid and evaporating the solution.—W. E. F. P.

**Roasting furnace.** F. Fouarge, Swansea, Eng. Pat. 102,595, Sept. 4, 1916. (Appl. No. 12,491 of 1916.)

THE furnace has superimposed hearths with openings at the ends so disposed that the material travels along a sinuous path. Each roasting chamber is provided with lateral shelves or flanges which do not extend quite to the ends of the chamber. Reciprocating rakes are carried by rods extending across the chambers and their ends slide upon the inwardly projecting flanges or below these. The rakes on alternate hearths work in opposite directions, pushing the material towards the shoot leading to the next lower floor as well as to one side. Dust-chambers communicating with the upper hearth are situated at the top of the furnace. Additional side shoots may be provided to prevent the channel from getting clogged.—W. R. S.

(A) **Furnace and allied structure.** (B) **Furnace.** (C) **Feeding device for furnaces.** (D) and (E) **Metallurgical furnace.** U. Wedge, Ardmore, Pa. U.S. Pats. (A) 1,208,246, (B) 1,208,247, (C) 1,208,248, (D) 1,208,249, and (E) 1,208,250. Dec. 12, 1916. Dates of appl., (A) Jan. 12, 1914, (B) Feb. 11, 1914, (C) Feb. 28, 1914, (D) Nov. 21, 1914, and (E) Mar. 1, 1915. All renewed Apr. 27, 1916.

(A, IN a furnace having a series of superposed annular hearths, each hearth is provided with an inner and an outer charge-receiving surface, side by side, each having a separate outlet. A charge is fed to each surface independently and moved over the surface towards the outlet. The outlets of alternate hearths are at the outer periphery and midway between the outer and inner peripheries respectively. (B) Two concentric cylindrical walls are provided with superposed annular hearths projecting into the annular space towards one another. A girder rotating in a horizontal plane about a central pivot carries depending from its outer end a series of annular hearths intermediate with the fixed hearths and projecting equally between the members of the two series. (C) In a furnace as in (B) the outer end of the girder carries a feeding hopper above a circular pivoted distributing plate over the hearths. The plate engages with fixed members on the furnace as the girder revolves, and is caused to rotate upon its own axis to distribute the

material (ore) on to the uppermost hearth from which it passes to the lower hearths. (D) In a metallurgical furnace a tubular vertical shaft is surrounded by a hearth having a treating chamber over it. A hollow rabble arm with a return passage therein communicates at one end with the tubular shaft and at the other end with a concentric tubular member within the shaft. An air distributing arm projects into the working chamber from one of the tubular members independently of the rabble arm. It is provided at its inner end with a valve to regulate the flow of air to the working chamber, the air being supplied to the other tubular member. (E) A rabble blade supported by its enlarged head in jaws projecting downwards from a hearth or equivalent support. A rib or flange on the support bears on the rear face of the rabble blade. A filling body occupies the space in the support above the rabble blade.—W. F. F.

*Metal articles; [Preventing] scaling, and annealing of*—. F. Perry, and Metalloids, Ltd., Tipton, Staffs. Eng. Pat. 102,660, Mar. 25, 1916. (Appl. No. 4445 of 1916.)

the treatment of metal articles in a muffle or other annealing chamber containing an atmosphere of Mond gas or similar gas for reducing or preventing the formation of scale, the gas is freed from water vapour, sulphur, and unsaturated and saturated hydrocarbons before admission to the muffle. The saturated hydrocarbons (e.g., methane) are eliminated by passing the gas through an iron tube, or a chamber containing loose pieces of iron, heated to about 250° C., after removal of the other impurities by known means.—W. E. F. P.

*Metals [copper] from ores; Process for the recovery of*—. C. S. Vadner, Butte, Mont. U.S. Pat. 1,207,243, Dec. 5, 1916. Date of appl., May 23, 1916.

The oxide or roasted sulphide ore mixed with a pot chloride solution is treated with sulphur dioxide. The clear liquor is freed from the excess of sulphur dioxide, partially neutralised, and iron and arsenic are precipitated by introducing air in presence of a carbonate. After completely neutralising, the copper is precipitated as cuprous chloride by heating. Sodium sulphate is recovered from the liquor.—W. R. S.

*Copper and nickel ores; Treatment of*—by electrolysis. V. Garin. Fr. Pat. 481,079, Feb. 29, 1916.

Albuminous or gelatinous materials, such as albumin, gelatin, dextrin, etc., are employed in the electrolytic deposition of copper and nickel from impure solutions, charged with sulphurous acid, particularly those obtained by the lixiviation of copper ores. Filaments of albumin, gelatin, etc., are applied by rubbers of soft fibrous wood, obtained from the cotton plant, coconut palm, etc., covered with organic material, such as skins, etc., in order to remove the hydrogen bubbles from the rotating cylindrical cathodes, or the albuminous or like material may be employed as pieces floating in the electrolyte. The formation of copper sulphide is prevented, and a sound and compact deposit is maintained, even up to the point at which the bath is exhausted.—B. N.

*Zinc, copper or other metals; Electrolytic production of*—. Soc. de Métallurgie Electrolytique. First Addition, dated Sept. 24, 1915, to Fr. Pat. 472,764, May 28, 1914 (this J., 1915, 497).

Several rotary mandrel-cathodes, of small diameter but great length and supported on bearings so that they may be easily removed, are arranged in a shallow rectangular tank, each cathode being partly surrounded by a perforated lead anode in

the form of an open trough. Adjacent anodes are bound together by rivets, and they rest on supports fixed to the bottom of the tank. Impregnated wiping pads, of soft supple animal membranes, prepared from skins, bladder, etc., rendered insoluble by immersion in aldehyde, acetone, etc., are employed to remove bubbles of hydrogen from the cylindrical cathodes, and the action of the gelatin, added to the bath to improve the deposit, is thus made more regular. The animal membranes may be replaced by vegetable materials impregnated with insoluble gelatin. The pads, resting lightly on the upper surface of the cathodes, are displaced by an irregular reciprocating motion. The electrolyte, filtering between the rotating shafts of the cathodes and their supports, is collected in a receiving vessel, and led off from the bottom of the latter, to prevent the liquid coming into contact with the turning mechanism.—B. N.

*Melting metals [copper, bronze, and cuprous metals] which fuse at high temperatures; Process for*— in iron or steel ladles. J. Cookerley, Inglenook, Ala., Assignor to E. Shelton, Birmingham, Ala. U.S. Pat. 1,208,717, Dec. 12, 1916. Date of appl., Feb. 21, 1916.

The metal is melted, in contact with a bath of fused sodium chloride, in an iron or steel ladle or crucible, the inner surface of which has been previously coated with the fused sodium chloride.—W. R. S.

*Brass surfaces; Process of crystallising*—. O. R. Hasenohr, Assignor to J. Debeck, Terre Haute, Ind. U.S. Pat. 1,207,910, Dec. 12, 1916. Date of appl., Oct. 14, 1914.

The brass is immersed for 5–10 secs. in a solution containing 1 part of nitric and 2 of sulphuric acid, then washed in cold water and immersed for 5–10 secs. in an aqueous solution of potassium cyanide (8 oz. per gall.), again washed in cold water and placed for 1–5 mins. in sawdust impregnated with a solution made by dissolving 10 oz. of ammonium chloride and 2 oz. of copper sulphate in  $\frac{1}{2}$  gall. of water, and subsequently washed in cold water and dried after immersion in boiling water.—W. E. F. P.

*Gold; Process of recovering*—. J. H. Alling, Columbia, Cal., Assignor to F. M. Wright, San Francisco, Cal. U.S. Pat. 1,207,261, Dec. 5, 1916. Date of appl., Oct. 18, 1910. Renewed Aug. 18, 1911.

The ore pulp is mixed with a solution of common salt, and a continuous supply of the mixture passed once only through a series of alternate electrolytic and non-electrolytic cells. The stream of mixture is thus repeatedly subjected to electrolytic action at intervals sufficiently long to permit combination of the gold with the liberated chlorine; the gold is deposited on the cathodes.—W. R. S.

*[Zinc] retorts; Method of recovering clay and slag from used*—. C. C. Conover and A. N. Detweiler, Springfield, Ill. U.S. Pat. 1,207,503, Dec. 5, 1916. Date of appl., June 27, 1916.

Old clay retorts, which have been used for zinc distillation, are coarsely crushed, and the powder is subjected to a weak magnetic power which separates the more magnetic constituents of the slag. The coarse powder, after re-crushing, is treated by strong magnetic power to separate the less magnetic constituents of the slag from the clay. The material is subjected to the process before the magnetic properties of the slag constituents have been materially altered through oxidation.—W. R. S.

**Zinc; Manufacture and condensation of volatile metals, more especially — in the electric furnace.** A/S Metallforedling. First Addition, dated Nov. 6, 1915, to Fr. Pat. 478,770, Jan. 27, 1915 (see Ger. Pats. 289,493, 290,499, 290,690, and 291,492 of 1914; this J., 1916, 427, 606, 695).

THE gaseous products from the treatment of the slag, as described in the main patent, are conducted into the condensation chambers, thus enriching the vapour in zinc and facilitating the condensation in liquid form.—B. N.

**Metal; Process of carbonising and hardening —.** N. H. Bray, San Francisco, Cal. U.S. Pat. 1,207,848, Dec. 12, 1916. Date of appl., Apr. 29, 1914.

METAL is heated to a high temperature by direct application of an oxyacetylene flame containing an excess of acetylene. Potassium cyanide and a solution of salt and copper sulphate are then applied to the metal.—W. F. P.

**Ores; Process of smelting metallic —.** H. L. Doherty, New York. U.S. Pat. 1,207,881, Dec. 12, 1916. Date of appl., June 22, 1909.

A MIXTURE of ore and flux is heated in a rotating chamber, by direct contact with a flame of combustible gas and preheated air, until combination is effected between the gangue and flux; it is then transferred to a rotating reducing chamber in which an atmosphere of carbon monoxide is maintained by the partial combustion of carbonaceous matter in aerial suspension. The charge is mixed with solid reducing material at the point of entry into the second chamber, in which the heating is continued until complete reduction and fusion are effected. The hot reducing gases from the second chamber are employed in admixture with preheated air for heating the first chamber.—W. E. F. P.

**Galvanising wire; Furnace for —.** G. L. Meaker, Joliet, Ill. U.S. Pat. 1,208,185, Dec. 12, 1916. Date of appl., June 27, 1913.

IN an apparatus having a fire-box at one end and a shallow container for the coating metal (zinc) at the other, the container is provided with a long side tube to serve as an annealing bath; this extends through a horizontal flue to above the fire-box and is then bent upwards to prevent escape of the molten metal and provide an outlet for the treated wire. One end of the horizontal flue communicates with the fire-box and the other with a flue beneath the container, the arrangement being such that the annealing bath is cooled externally by a downward current of air at that part adjoining the container, and heated most strongly at the exit end.—W. E. F. P.

**Tinning, leading, and galvanising [small articles]; Mechanical process for —.** C. Ziegler. Fr. Pat. 479,970, Oct. 8, 1915.

THE articles to be coated are placed in or upon a perforated vessel or horizontal grid support adapted to be rotated about a vertical axis and, after immersion in the molten metal, drained and whirled to remove excess of the latter. The coated articles are then separated from each other before solidification of the coating metal occurs.—W. E. F. P.

**Metals; Method of separating fumable — [from alloys] by fractional distillation.** J. Thomson, New York, Assignor to John Thomson Press Co., Jersey City, N.Y. U.S. Pat. 1,208,237, Dec. 12, 1916. Date of appl., Feb. 8, 1916.

THE alloy is heated in one compartment of a container to eliminate the metal having the lowest vaporising point; the residue is displaced by additional charges of alloy, and made to flow

into an adjacent compartment where it is vaporised by a higher temperature. The fumes of the different metals are condensed separately.—W. E. F. P.

**Soldering composition.** H. Hess. Fr. Pat. 480,187, Nov. 11, 1915. Under Int. Conv., Oct. 15, 1915.

A PASTE composed of soldering metal powder 85-646—90-07, ammonium chloride 2-53—2-55, glycerin 5-43—5-85, and glue (aqueous) 0-59—1-16%. The glue is prepared by soaking 1 part of glue in 2 parts of water, heating to about 50°C, and mixing the jelly with  $\frac{1}{4}$  times its quantity of glycerin.—W. E. F. P.

**Bronze powder; Process of manufacturing —.** M. J. Fuchs. Fr. Pat. 480,504, Dec. 23, 1915.

THE coarse alloy is pulverised by stamping in a mill consisting of a series of compartments arranged at successively lower levels. The weight of the stamps decreases in successive compartments, and in each of the latter the dies or anvils are arranged in steps, down which the charge is caused to travel by the vibration produced.—W. E. F. P.

**Metal [aluminium]; Manufacture of powdered, granulated —.** H. J. Jack and A. G. Lohley. Fr. Pat. 480,563, Dec. 29, 1915. Under Int. Conv., Dec. 6, 1915.

A VERTICAL stream of the molten metal is subjected to the action of a horizontal blast of air or other gas under high pressure, whereby subdivision and solidification of the material is effected.—W. E. F. P.

**Tungsten or other analogous metal [for incandescent filaments]; Process and [electric] fusion furnace for the manufacture of coherent ductile —.** Soc. Anon. ci-dev. Gmur et Cie. Fr. Pat. 480,819, Jan. 31, 1916. Under Int. Conv. Jan. 15 and July 11, 1914.

COHERENT ductile tungsten is obtained by fusing the metal, and then rapidly cooling by directing a current of air on to the crucible containing it. The fusion furnace is composed of a crucible resting on a fixed base-plate forming one electrode and surrounded by two concentric heating jackets connected to a common electrode. The tuyers for the cold air are arranged outside the heating jackets along the whole length of the crucible and within an external insulating jacket. The heating jackets may be raised out of the furnace by a commutator, bound to the lifting arrangement automatically cutting off the current at the same time, whilst the valve for the air is opened simultaneously.—B. N.

**Bronze; New —.** E. A. de Lisle and N. A. Hélois. Fr. Pat. 480,928, May 5, 1915.

COPPER-VANADIUM alloy, containing 4 to 5% of vanadium, reduced from the oxides by aluminium is mixed with electrolytic copper and aluminium to give an alloy containing 0.1 to 0.3% of vanadium and 1% of aluminium, and, when melted, 4% of aluminium is added. The copper-vanadium may be replaced by copper-uranium, or preferably copper-uranium-vanadium, so as to introduce into the copper, 5 to 7% of aluminium, 0.1 to 0.3% of vanadium, and 0.2 to 0.4% of uranium; a hard resistant alloy is produced, capable of taking a high polish.—B. N.

**Metals [aluminium and silicon]; Process of apparatus for obtaining — [from clay, etc.]** Weaver Co. Fr. Pat. 481,056, Feb. 25, 1916.

A MIXTURE of clay, etc., with carbon is heated in an electric furnace in a stream of chlorine and volatilised aluminium and silicon chlorides are separated by fractional condensation, all the



operations being conducted in an atmosphere free from moisture. The silicon tetrachloride produced is decomposed by intimate contact with molten aluminium with formation of aluminium chloride and silicon; the aluminium chloride is electrolysed in molten sodium chloride, the chlorine liberated in the latter operation being used for treating a further quantity of clay. The process is continuous, the apparatus consisting of a closed system comprising furnace, condenser, receiver, electrolytic vat, and gas reservoir (for chlorine) connected in series, the gas reservoir also communicating with the furnace inlet. The condenser consists of two parts adapted to be cooled to different temperatures; and the interior of the receiver is maintained above atmospheric pressure.—W. E. F. P.

*Zinc; Extraction of* —. E. S. Berglund. Fr. Pat. 480,633, Jan. 7, 1916. Under Int. Conv., May 31, 1915.

IN a process for smelting zinc ores by electrical means, the bulk of the zinc vapour is condensed as liquid metal and the remainder as zinc dust which is subsequently volatilised out of contact with air and re-condensed in the same system. The condenser has two communicating compartments, the first connected with the outlet and the second with the inlet of the electric furnace. Liquid metal is condensed in the first compartment, zinc dust being deposited in the second and conducted therefrom to the interior of the furnace, out of contact with air, by means of a screw conveyor.—W. E. F. P.

*Furnaces; Tilting or rolling* —. D. F. Campbell. London, and Soc. Electro-Métallurgique Française, Froges, France. Eng. Pat. 102,902, Aug. 9, 1916. (Appl. No. 11,250 of 1916.)

*Welding composition*. J. A. Hope, Montréal, Canada. U.S. Pat. 1,209,841, Dec. 26, 1916. Date of appl., Apr. 10, 1916.

SEE Eng. Pat. 102,237 of 1916; this J., 1917, 38.

*Iron from scrap of zincd sheet iron; Process of manufacturing* —. O. Schleimer, Assignor to K. Albert Chem. Fabr., Neuss, Germany. U.S. Pat. 1,209,628, Dec. 19, 1916. Date of appl., Nov. 13, 1914.

SEE Ger. Pat. 280,414 of 1913; this J., 1915, 497.

*Alloy for arc welding*. D. H. Wilson, Franklin Township, N.J., and S. Rodgers, Pittsburgh, Pa., U.S.A. Eng. Pat. 18,115, Dec. 29, 1915. Under Int. Conv., June 16, 1915.

SEE U.S. Pats. 1,187,411 and 1,187,412 of 1916; this J., 1916, 849.

*Copper; Extraction of* —. Metals Research Co., Assignees of E. R. Weidlein, Thompson, Nev., U.S.A. Eng. Pat. 101,339, Aug. 2, 1916. Under Int. Conv., Aug. 31, 1915. (Appl. No. 10,933 of 1916.)

SEE U.S. Pat. 1,201,899 of 1916; this J., 1916, 1223.

*Zinc; Electrolytic recovery of* — from ores and other zinc-bearing materials. U. C. Tainton, Johannesburg, S. Africa. U.S. Pat. 1,210,017, Dec. 26, 1916. Date of appl., Aug. 10, 1915.

SEE Eng. Pat. 11,335 of 1915; this J., 1916, 969.

*Roasting-furnace; Mechanical* —. W. Schefczik, Hamburg, Germany. U.S. Pat. 1,210,852, Jan. 2, 1917. Date of appl., May 22, 1914.

SEE Fr. Pat. 473,310 of 1914; this J., 1915, 557.

*Crucible furnace; Recuperative* — for fusion of metals and alloys. A. Hermansen. Fr. Pat. 480,636, Jan. 8, 1916.

SEE Eng. Pat. 15,887 of 1915; this J., 1917, 36.

*Tin; Extraction of* — from cassiterite and from oxidation products of tin. G. Michaud and E. Delasson. Fr. Pat. 481,062, Dec. 10, 1915. Under Int. Conv., Jan. 14, 1915.

SEE Eng. Pat. 614 of 1915; this J., 1916, 314.

*Production of a thick or compact metallic coating on artificial stones and articles of artificial stone mass*. Eng. Pat. 17,864. See IX.

*Treatment of waste tins in a dust destructor*. Eng. Pat. 102,693. See XIXB.

## XI.—ELECTRO-CHEMISTRY.

*Overvoltage tables. Part IV. The theories of overvoltage and passivity*. E. Newbery. Chem. Soc. Trans., 1916, 109, 1359—1368. (See this J., 1916, 1265; 1917, 38.)

ELEMENTS in the same group of the periodic system show the same overvoltage, and when the overvoltage of a metal changes in such a way as to correspond with the typical overvoltage of another group, compounds of that metal are generally known in which the metal has the valency characteristic of the new group. Overvoltage is therefore stated to be probably due to the high solution potentials of compounds of the electrode material with the discharged ion, or with a product of the discharged ion. These compounds tend to form solid solutions in the electrode substance, and are usually stable only under the influence of high pressures or high temperatures. A slight fall of overvoltage is probably due to physical forces, such as the inductive action of escaping ionised gas at high current densities, whilst greater diminution of the overvoltage is more probably due to chemical forces, resulting in the breakage of the electrode surface, the release of internal pressures, and subsequent decomposition of the compounds present. It is probable that compounds of the nature described play a considerable part in the production of thermionic currents. Passivity is stated to be due to the insolubility and good electrical conductivity of the above compounds, which form a protective coating either over the whole surface or over the more easily soluble amorphous phase only. The coating may consist of the pure compound, or of a solid solution of the compound in the electrode substance. The chemical stability of the higher oxides or hydrides formed on the surface of the electrode is of greater importance than the overvoltage, when dealing with substances which are only slowly oxidised or reduced.—B. N.

*Sulphuric acid and normal potassium sulphate; Ionisation of* — in aqueous solutions of medium concentration. J. A. Muller. Bull. Soc. Chim., 1916, 19, 438—441.

By the electrolysis of dilute sulphuric acid in a divided cell in which the anode was surrounded by a solution of barium chloride, it was shown that the amount of barium sulphate formed in the anode compartment compared with the hydrogen liberated at the cathode corresponded to a molecule of the former to 2 atoms of the latter. This points to the ionisation of the sulphuric acid at the dilution employed (2N) into  $\text{SO}_4^{--}$  and  $2\text{H}^+$ , and not into  $\text{SO}_4\text{H}^+$  and  $\text{H}^+$ , since in this latter case the formation of 1 mol. of barium sulphate, according to the equation,  $\text{SO}_4\text{H}^+ + \text{BaCl}_2 =$

$\text{HCl} + \text{BaSO}_4 + \text{Cl}$  would be accompanied by the liberation of only 1 atom of hydrogen, unless the complete electrolysis of the hydrogen chloride were postulated, which however was shown not to be the case by suspending barium carbonate in the anodic solution in a second experiment, when although no free hydrochloric acid could be formed, still the same volume of hydrogen was liberated at the cathode. By a similar method potassium sulphate was shown to be ionised into  $\text{SO}_4$  and  $\text{K}$ , to the exclusion of  $\text{KSO}_4$  ions.

—G. F. M.

*Electrolyses; Remarks on certain*—J. A. Muller. *Bull. Soc. Chim.*, 1916, 19, 411—414.

ALTHOUGH in the electrolysis of the system sulphuric acid, barium chloride (see preceding abstract), no barium passes into the cathode compartment, and sulphuric ions equivalent to all the hydrogen liberated are found in the anode compartment, when potassium hydroxide is substituted for the barium chloride a complete interchange of ions is observed, and a considerable difference exists between the hydrogen liberated and that corresponding to the migration of potassium and sulphate ions; this is attributed to an ionisation of the hydroxide into both  $\text{K}$  and  $\text{OH}$  and also  $\text{H}$  and  $\text{OK}$  ions. The electrolysis of the system sulphuric acid, hydrochloric acid, gives chlorine at the anode until the concentration of the hydrochloric acid falls below  $N/100$ , when principally oxygen is liberated. In this case the discrepancy between the observed and calculated volumes of hydrogen liberated is due to the formation of oxygen compounds of chlorine. When a molar solution of auric chloride is placed in the anode compartment, with sulphuric acid in the cathode compartment, neither chlorine nor gold is liberated at the electrodes, and the process resolves itself into an electrolysis of water, catalysed by the auric chloride. This may be due to the formation, decomposition, and re-formation of small quantities of hydroxychlorides of the type  $\text{AuCl}(\text{OH})_x$ . It is otherwise when a solution of auric chloride alone is electrolysed; in this case gold is deposited and, at the commencement, ozonised oxygen liberated at the anode, accompanied later by chlorine as soon as the concentration of free hydrogen chloride exceeds  $N/100$ .

—G. F. M.

*Synthesis of cyanides in the electric pressure-furnace.* Stähler. See VII.

*Catalysis of hydrogen-oxygen mixtures at the ordinary temperature by moist contact substances.* Hofmann and Ebert. See VII.

#### PATENTS.

*Electrolytic apparatus.* R. L. Whitehead, Perth Amboy, N.J., Assignor to American Smelting and Refining Co., Maurer. N.J. U.S. Pats. (A) 1,206,963, and (B) 1,206,965, Dec. 5, 1916. Dates of appl., (A) Feb. 10, 1913 and (B) Apr. 17, 1916.

(A) ELECTRODES are suspended from a bar of conducting material which extends across an electrolytic refining tank, the bar being supported at the ends by suitable members. Contact is made between these members and the bar, and between the bar and electrodes, or between electrodes, by means of a ridge on the one and a recess on the other, the walls of the recess coming into wedging contact with the sides of the ridge. (B) Several electrolytic vats are each provided with sets of anodes and cathodes, the set of anodes having lateral projections for supporting them on

the walls of the vat, whilst the cathodes are each provided with a lateral projection extending beyond the wall of the vat, and making direct contact with an anode in an adjacent vat by bearing thereon by gravity. The set of cathodes may be raised vertically out of the vat, and thus disconnected electrically from the anodes.—B. N.

*Separator for storage batteries.* W. L. Bliss, Assignor to U.S. Light and Heat Corporation, Niagara Falls, N.Y. U.S. Pat. 1,206,983, Dec. 5, 1916. Date of appl., Mar. 27, 1914.

A FINELY-DIVIDED acid-resisting material, such as asbestos fibre, and a finely-divided soluble material, are mixed with a phenolic condensation product, such as bakelite. The mass is hardened under the combined action of heat and pressure, and treated to dissolve the soluble material.—B. N.

*Electrolytic cell.* D. Shaw, New York. U.S. Pat. 1,208,722, Dec. 12, 1916. Date of appl., June 22, 1915. Renewed Oct. 11, 1916.

A RECEPTACLE, forming an electrode, is provided with a flange extending laterally from its upper edge; a support, with a central opening closed by a cover plate, is mounted upon the receptacle serving partly to close it, and the support is secured by suitable means to the flange. A sleeve, depending from the edge of the opening of the support, is secured to the top of a porous bag-shaped flexible diaphragm, and an electrode secured to the cover but insulated therefrom, is disposed within the diaphragm. Both the support and the cover plate are provided with gas outlets, for conducting gas from the interior of the receptacle externally of the diaphragm, and from the interior of the diaphragm respectively.—B. N.

*Furnaces; Combined plug and cooler for electric arc working under high pressure.* Norsk Hydro-Elektrisk Kvaelfakteselskab. Fr. Pat. 480,302, Nov. 23, 1915. Under Int. Conv., Dec. 10, 1914.

IN electric arc furnaces working under a high pressure, a hollow plug is attached directly to the outlet tube of the furnace, and is connected with an arrangement for producing an external pressure, and for cooling. For example, the plug may form the flame tube of a steam boiler, whilst in an alternative arrangement, the upper part of the furnace is cooled by a water jacket connected to, and serving as a preheater for, the boiler.—B. N.

*Resistance of carbon electrodes; Reducing the—* Soc. Franç. des Electrodes. Fr. Pat. 480,806. May 3, 1915.

THE resistance of carbon electrodes is diminished by incorporating with the electrode material powdered metals, such as copper, aluminium, etc., or metallic oxides and salts which will yield nascent metals under determined conditions, or natural mineral substances, such as graphite, or a mixture of these.—B. N.

*Electrode for electrolytic purposes; Wire—* M. Huth, Charlottenburg, Assignor to Siemens und Halske A.-G., Berlin. U.S. Pat. 1,209,710, Dec. 26, 1916. Date of appl., July 7, 1916.

SEE Eng. Pat. 100,730 of 1916; this J., 1916, 981.

*Electrolytic cells for the decomposition of solutions.* C. J. (1914) Syndicate, Ltd. Fr. Pat. 480,127. Nov. 4, 1915. Under Int. Conv., Nov. 6, 1913, and Nov. 21, 1914.

SEE Eng. Pats. 25,415 of 1913 and 22,867 of 1914; this J., 1914, 1096; 1916, 55.

*Electrolytic manufacture of aromatic amino-[hydr]-ory compounds.* Eng. Pat. 18,081. See III.

*Electrolytic manufacture of sodium perborate.* Eng. Pat. 100,154 and 101,620. See VII.

*Direct process for the manufacture of ammonium chloride from chlorine, nitrogen, and hydrogen.* Fr. Pat. 480,232. See VII.

*Process and apparatus for softening water.* Fr. Pat. 480,408. See XIXb.

## XII.—FATS; OILS; WAXES.

*Olive pomace: Utilisation of —.* W. V. Cruess and A. W. Christie. J. Ind. Eng. Chem., 1917, 9, 45—47.

It is estimated that about 4000 tons of residue ("pomace") are left each year after expression of the oil from olives in California, and at present most of this is wasted. Pomace from eighteen olive factories contained from 7.89 to 20.23%, or 20.98 to 53.81 galls. of oil per ton of the fresh product. This oil could be best extracted by our extractions with petroleum spirit (gasoline), the minimum yield from air-dried pomace being 5.5 galls. per ton. Oils thus extracted from two samples had sp. gr. 0.903 and 0.890 and saponification value 173.8 and 163.2, and contained 11.77 and 64.63% of free acids (as oleic acid) respectively. For soap-making they compared favourably with pure olive oil. Distillation by direct heat removed practically the whole of the solvent from the residual pomace, but it could not be completely separated by distillation with steam. Air-dried samples of the fresh and extracted pomace contained:—Potassium oxide, 0.24 and 0.26; phosphoric acid ( $P_2O_5$ ), 0.12 and 0.14; and nitrogen 0.86 and 1.00% respectively. The nitrogen was shown by practical tests to be "unavailable," whilst the amounts of potash and phosphoric acid were no greater than are present in average California soils. Hence the pomace is of no commercial value as a fertiliser.—C. A. M.

*Utilisation of cherry by-products.* Rabak. See XIXa.

*Combustion methods for use in the laboratory.* [Determination of sulphur.] Hewett. See XXIII.

## PATENTS.

*Oil from whale blubber; Process and apparatus for extracting —.* Myrens Verksted. Fr. Pat. 479,962, Oct. 8, 1915. Under Int. Conv., Oct. 10, 1914.

WHALE blubber, preferably cut into small pieces, is heated for a short time to coagulate and separate the blood, prior to the expression of the oil. The heating is effected partly by direct and partly by indirect means, to prevent condensation of the steam. For this purpose the vessel is provided with two series of steam conduits, one of which is perforated to allow the steam to escape into the interior of the vessel, whilst in the other the steam is not allowed to escape. After heating, the blubber is conveyed to a press by means of a screw conveyor or the like.—C. A. M.

*Oils and fats; Apparatus for purifying industrial —.* L. J. Noël, Epemay, France. U.S. Pat. 1,209,480, Dec. 10, 1916. Date of appl. May 14, 1914.

SEE Fr. Pat. 458,049 of 1913; this J., 1013, 1021.

*Preparation of solutions of vulcanised oils, obtained by the action of sulphur chloride, in amyl acetate.* Fr. Pat. 480,939. See XIII.

*Process of making [fatty] food products.* U.S. Pat. 1,206,954. See XIXa.

*Disinfectant, antiseptic, deodorant, and detergent materials.* Eng. Pat. 16,410. See XIXb.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Copying ink pencils and the examination of their pigments in writing.* C. A. Mitchell. Analyst. 1917, 42, 3—11.

THE following table gives the composition of the pigments of representative copying-ink pencils as sold from 1907 to the present time:—

	Description.	Origin.	Date.	Moisture (Loss at 100° C.)	Residue insoluble in alcohol	Dyestuff.	Loss on ignition (Graphite, etc.).	Ash (Kaolin, etc.).
				%	%	%	%	%
1	American Pencil Co., Duplex, Blue . . . . .	U.S.A.	1911	2.35	76.45	21.20	27.09	47.36
2	American Pencil Co., Duplex, Violet . . . . .	U.S.A.	1916	3.96	59.28	36.76	27.36	31.92
3	American Pencil Co., "Ink Kau," No. 160 . . . . .	U.S.A.	1916	7.48	51.53	40.99	25.48	26.36
4	American Pencil Co., Venus, No. 167 . . . . .	U.S.A.	1916	2.39	60.89	36.81	56.54	4.06
5	Eagle Pencil Co., Atlas, No. 823 . . . . .	London	1907	4.85	67.77	27.70	11.66	50.11
6	Eagle Pencil Co., Copying Eagle Ink . . . . .	U.S.A.	1911	3.75	63.95	32.30	13.90	50.06
7	Eagle Pencil Co., Copying Eagle Ink, No. 824 . . . . .	U.S.A.	1911	3.75	63.95	32.30	13.90	50.06
8	Eagle Pencil Co., Leads, No. 119 . . . . .	U.S.A.	1911	3.55	57.14	36.84	23.18	58.86
9	Faber Copying . . . . .	Bavaria	1907	3.72	64.48	31.80	11.66	47.77
10	Faber Copying . . . . .	Bavaria	1911	2.84	71.27	26.00	29.54	29.84
11	Faber Blue, No. 2251 . . . . .	Bavaria	1907	1.82	58.68	49.94	44.14	3.00
12	Hardtmuth "Mephisto" . . . . .	Austria	1907	3.54	47.14	33.24	68.12	4.80
13	Hessin, No. 74 . . . . .	U.S.A.	1911	3.74	72.92	23.64	52.64	8.76
14	"Ink and Copying" . . . . .	U.S.A.	1911	5.06	61.49	26.60	40.61	17.68
15	Kutz . . . . .	Bavaria	1907	3.83	57.59	38.68	64.86	4.50
16	"Lapis Titius" . . . . .	—	1911	2.04	69.86	22.42	69.17	3.66
17	Rowney . . . . .	Britain	1911	4.53	73.05	35.84	7.61	52.22
18	Swan Copying, No. 1039 . . . . .	Bavaria	1907	6.33	59.83	51.52	5.50	40.98
19	Swan Copying, No. 1030 . . . . .	Bavaria	1911	2.96	46.43	31.80	62.08	3.40
20	Wolff's "British Preference" . . . . .	London	1916	2.72	65.48	32.74	62.56	2.79
21	Wolff's "Royal Sovereign" . . . . .	London	1916	2.91	65.35	32.74	62.56	2.79

The dyestuff was determined by treating the dried powdered pigment with successive small quantities of warm 85% alcohol, and drying the insoluble residue in the water oven. In all the pigments of violet colour the extracted dyestuff gave the reactions of Methyl Violet. The soluble blue pigment in No. 1 was an aniline dyestuff, whilst the pigment of No. 11 contained Prussian blue and Methyl Violet. As part of the ash is derived from impurities in the graphite it is only possible to make an approximate estimation of the relative proportions of graphite and kaolin from the analyses. Impure graphite may contain only about 50 to 70% of carbon, but the better kinds used for pencils generally contain not more than 3 to 4% of impurities (iron oxide, silica, etc.). When the ash is less than 5% (Nos. 4, 12, 13, 16, 17, 20, and 21), it is probable that the mineral matter was derived solely from the graphite. The ash of No. 14 (8.76%), consisting largely of ferric oxide, was also probably due to the graphite. By attributing about 5% of the mineral matter (calculated on the original pigment) to the graphite, and adding this to the loss on ignition, a result approximating to the proportion of impure graphite originally used would be obtained. These pigments may therefore be classified into four main groups, viz.: (1) Those composed of graphite and dyestuff only; (2) those in which the dyestuff has been mixed with approximately equal proportions of graphite and kaolin (Nos. 2, 3, 5, and 8); (3) those in which the kaolin is largely in excess (Nos. 1, 6, 7, 9, 10, 18, 19); and (4) one case (No. 15) where the graphite predominates. Some of the pigments contained alumina (0.59% in No. 3, to 10.37% in No. 18). This affected the extraction of the dyestuff with alcohol, and caused the pigments containing it to give poor copies in copying tests. The copying properties appear to depend on the proportion of the dyestuff, and on the nature of the graphite, as well as on the character of the kaolin. The best copying results were obtained with No. 3 "Ink Eau" and No. 12, Hardtmuth's; the worst with No. 16 "Lapis Tinta" and Nos. 18 and 19 (Swan). The behaviour of the written characters on treatment with a drop of water, or, in other words, their copying properties in miniature, may be used as a means of differentiating between the pigments in writing. Solution tests with drops of acetic acid and of ether may also be applied to the writing, whilst chemical tests with nitric acid and various reducing and oxidising agents will also enable the writings done with the different pigments to be distinguished. A further test with potassium ferrocyanide solution depends on the fact that the iron oxide in these pigments may range from nil to upwards of a third of the total ash. Writing done with these pencils upon wood was sufficiently permanent to resist immersion for over two months in strong salt water, which was frequently shaken and exposed to sunlight.—C. A. M.

*Resin; Sources of — in Germany.* [Artificial seasoning of wood.] E. R. Besenfelder. Chem.-Zeit., 1916, 40, 997—1000.

THE total production of utilisable conifer wood in 1913 in Germany is estimated at 12,462,000 metric tons, whilst 5,840,563 tons of resinous wood in various forms was imported. By the general adoption of the rapid method of seasoning timber, and by the extraction of sawdust, waste wood, etc., it would be possible to render Germany independent of imported rosin and oil of turpentine. In Schilde's rapid seasoning process (*Der Holzmarkt*, 1916, No. 124) the freshly-sawn timber is heated in a closed vessel and subjected to the action of a steady current of vapour from an organic liquid, preferably a good solvent for resins and fats. This causes the sap to exude from the

wood, and while part falls as liquid to the bottom of the vessel, the remainder passes with the vaporised solvent to a condenser, where the aqueous portion separates and is drawn off. The condensed solvent, charged with rosin, turpentine, fat, etc., is conducted to an evaporator, whence the vapours are returned to the seasoning vessel. The drying process is continued until the water only separates in drops, and it is essential that the solvent should not be allowed to condense within the pores of the wood, since this affects its firmness. So long as moisture is present in the wood the temperature in the vessel keeps below the boiling point of the lower boiling constituent, e.g., at 75° C. when the mixture consists of water and trichlorethylene (b.p. 88 to 90° C.). After removing the last traces of solvent, and cooling the wood, which is quite free from cracks, is completely seasoned and sterile, and ready for immediate use for polishing, etc. The process entirely prevents the blue discoloration which is responsible for a considerable loss of timber in the ordinary seasoning process. The extract from the wood varies with the origin, species, and age of the tree. On the average fir yields about 1% of rosin, oil, etc. The thinner the planks of wood the greater the proportion of rosin obtained and *vice versa*. The value of this extract is estimated at not less than M. 1.40 per kilo. (about 7½d. per lb.) Taking it as only M. 1 per kilo. (about 5½d. per lb.), the average annual yield of resinous extract from German-grown wood would be worth M. 124,620,000 (about £6,000,000), while the imported wood would give an extract worth M. 46,225,700 (about £2,250,000). About half of the extract would consist of rosin and turpentine, and the remainder (120,200 metric tons) of fat, mainly compounds of oleic acid. This quantity of fat would be nearly a fourth of that contained in the oil-fruits imported into Germany. Sawdust, waste wood, etc., extracted by this process must be treated as soon as possible, since the solubility of the rosin rapidly decreases on oxidation. The extracted sawdust is particularly suitable for packing purposes and may be used as a filling material for fodder. The extracted material is suitable for making paper pulp, and may also be advantageously used for dry distillation, and has the advantage of yielding a strong pyrolygneous acid. It should also be sufficiently pure for nitration.—C. A. M.

*Varnish analysis and varnish control. II. Viscosity of varnishes.* M. Y. Seaton, E. J. Probeck, and G. B. Sawyer. J. Ind. Eng. Chem., 1917, 9, 35—40.

FOR the routine examination of varnishes the use of the Doolittle viscometer is recommended, the results being expressed in "degrees of retardation" in the rotation of a standard cylinder immersed in the varnish at 40° C. In the case of varnishes viscous at that temperature, smaller cylinders of ½ to 1 in. are substituted for the standard 1½ in. cylinder, the results being brought to the standard viscosities by reference to curve showing the relationship between the different cylinders. The two types of solution (true and colloidal) which appear to be present in all varnishes differ materially in their viscosity characteristics. The colloids also show pronounced differences, the "suspensions" having a viscosity but little higher than that of the dispersion agent, whereas the "emulsoids" may show a very high viscosity. The changes which occur in the viscosity of varnishes during ageing or on the addition of certain solvents can usually be traced to changes in the character of the colloids present. The colloidal material in varnishes is generally present in the emulsoid form and hence shows great variations in viscosity when the concentra-

of the varnish is increased, or when the degree of dispersion of the colloid is changed. In the case of varnishes of the true solution type, *e.g.*, those containing much soluble gum and but little polymerised oil, the curves showing the changes in viscosity with the temperature from 20° to 50° C. were very similar in form and quite distinct from the viscosity-temperature curves of varnishes of the colloidal type, which were practically straight lines. Hence by plotting the results obtained at different temperatures it is often possible to obtain information as to the nature of the varnish. As a rule the viscosity of emulsoids decreases on decreasing the degree of dispersion, and *vice versa*. Hence on adding a suitable solvent, such as rosin acids or acid linseed oil, to a varnish containing an emulsoid, such as polymerised wood oil, the viscosity should be lowered, and in practice this is found to be the case. In like manner, the addition of an active thinner to a colloidal varnish will change the viscosity-temperature graph from a straight line to a curve. The changes which are used by the degree of dispersion of the colloidal material in varnish are seen in the case of a black king Japan varnish, which, when incorrectly diluted, increased in viscosity from 83 to 110 in a month. The viscosity was reduced by the addition of rosin oil, which was a solvent for the asphalt, the actual change in the degree of dispersion of which caused the increase in the viscosity. By altering the method of manufacture a product was obtained, the viscosity of which fell from 91 to 74 in a month, and then remained constant, showing the change even when additional solvent was introduced. The method is also applicable to the investigation of lithographic linseed oils. One sample, for example, with an original viscosity of 69, gave a reading of 69 after a month, whereas a different type of oil increased in viscosity from 68 to 80 in the same time. The second oil was considered less valuable as a medium for paints. In general it has been found that with stable varnishes the viscosity becomes approximately constant after a month. If the viscosity continues to rise materially after that time it will usually be found that the increase will continue for a long time, until, finally, the varnish becomes unsuitable for general use.—C. A. M.

*linoleum; Manufacture of — and its valuation.*  
A. de Waele. J. Ind. Eng. Chem., 1917, 9, 6—18.

PART from the tests used by the German Government (this J., 1900, 255), and certain tests devised by Ingie (this J., 1904, 1197), there have been few chemical tests applied to linoleum. With regard to the determination of the "acetone extract" and "unsaponifiable matter insoluble in acetone," which appear in specifications for goods to be supplied to the English Government, it is pointed out that acetone, like ether, will extract an indefinite amount of soluble matter, and that the amount of "unsaponifiable matter" will vary with the conditions of saponification. For example, cork when saponified with alcoholic potash in an open basin yielded 33 to 36% unsaponifiable matter, whereas under a reflux condenser it yielded 72%. It is possible, however, to obtain constant results by saponification with 1.25% aqueous sodium hydroxide solution under reflux condenser, and the amount of unsaponifiable matter multiplied by 1.43 approximates closely to the amount of cork. *Manufacture of linoleum*:—Taylor-Parnacott or Corticine floor coverings differ from true linoleum in the fact that the oil is prepared by a polymerisation as distinct from an oxidation process. "Taylor cloth" or "cork carpet," is hung for several weeks in a steam-heated room at 120° to 160° F. (49°—71° C.), to convert the coating from the "green"

condition into a leathery product. In the Walton processes for linoleum (see Reid, this J., 1896, 75) the linseed oil is oxidised by boiling with suitable driers and pouring in thin successive layers over cotton fabrics or "scrims," or the raw oil is mixed cold with a small amount of insoluble drier and made to fall from a "shower bath" in a continual rain on to the bottom of an inclosed vessel heated by means of steam pipes, the thickened oil being afterwards mixed with 5% of whitening and "smacked" in horizontal steam-jacketed drums containing a horizontal shaft with radial arms. In the Wood-Bedford process the whole of the oxidation is effected in the "smacker" within 48 hours at temperatures up to 140° F. (60° C.), but owing to the speed of the reaction the product is greatly inferior to those obtained by the Walton processes. The oxidised oil, by whichever process obtained, is agitated in a steam-heated pan, while first an eighth of its quantity of melted rosin and then the same amount of gum kauri is run in, and the agitation is continued until the mixture thickens to the required degree, when the hot "cement" is poured on to revolving water-cooled rolls or other cooling device, and, after cooling, is allowed to "hang" or mature for a week or two. *Reactions*:—In the equations suggested by Ingie (this J., 1913, 640) for the change produced in linolenin, the position given to the double bonds is not in accordance with the commonly accepted structure for that compound, but there is evidence, in the evolution of volatile substances with aldehydic groupings, for the view that the reaction is one of rupture in the chain in the middle of a peroxide group. In the author's opinion the change is not one of polymerisation, but either a molecular rearrangement of complex groupings or a condensation or both. *Action of solvents on oxidised oils* (see this J., 1892, 550):—On extracting with ether a sample of the dried skin obtained by the scrim process, 67.50% was extracted in 24 hours, but the extraction was not complete after 41 months, when the extract was 85.32%. Similar results were obtained with alcohol, benzene, chloroform, and acetone, but petroleum spirit of b.p. 35° to 50° C., although also showing no definite end-point in the extraction, yielded extracts which differed in physical and chemical properties from the extracts obtained with other solvents. For the practical examination of these products two extractions give useful results:—(1) Extraction for 24 hours with petroleum spirit, and (2) extraction of the residue with ether. The petroleum spirit extract is a pale greenish-yellow, fairly mobile oil, which thickens but does not form a skin on exposure, whilst the subsequent ethereal extract is a viscous sticky yellowish-brown oil which dries on exposure, forming a fairly hard elastic skin. Taking "linoxyn" to represent the final insoluble residue, the variations in the distribution of the fractions in the "skins" obtained by the "scrim process" were as follows:—

	1	2	3	4	5	6	7
Linoxyn %	49.57	47.08	43.62	48.41	49.22	57.09	42.46
Ether extract %	31.16	26.79	27.93	23.38	25.45	25.16	32.91
Petroleum spirit extract %	19.27	26.13	26.45	22.21	25.33	17.75	24.63

It was proved that the physical properties of the "skins" were better in proportion to the amount of ether extract, and inversely so to the amount of petroleum spirit extract, whilst the residual linoxyn affected the "body" or solidity of the mass. On this basis No. 6 would be classed a

poor sample, whilst No. 7, in which the linseed oil is oxidised with a special drier, would be considered an excellent one, as in practice proved to be the case. In similar tests with oxidised oils prepared by the Walton "shower-bath" process, the following results were obtained:—

		1	2	3	4	5	6
Linoxyn .....	%	40.2	32.4	50.0	42.2	37.4	37.7
Ether extract ..	%	35.0	33.1	30.3	31.2	29.6	34.9
Petroleum spirit extract .....	%	24.7	27.7	18.3	26.3	33.0	26.4

The higher ether extracts of these products compared with those made by the "scrim" process account for their superior tenacity and binding power, and explain why they alone are suitable for the manufacture of "Walton inlaid." The following results obtained with Taylor-Parnacott or Corticine oil show why it is impossible to use that product for making inlaid linoleum and also explain its low covering power:—Linoxyn, 60.4; ether extract, 9.6; and petroleum spirit extract, 30.0%. *Linoxyn and "cyclolin"*:—The substance insoluble in ether which is obtained by polymerising linseed oil by heat has been provisionally termed "cyclolin." It differs in a pronounced way from the linoxyn produced in the oxidation of linseed oil. Thus its saponification value does not exceed that of the raw oil, it is difficult to saponify, and is very stable, whilst it contains no oxidised acid. Again, linoxyn is completely soluble in amyl alcohol at 120° C. (6 to 8 hours), whereas cyclolin swells up but only dissolves to the extent of about 20% in that solvent. Similar experiments with other oxidised oils and with polymerised wood oil showed that as a general rule oxidised oils are completely soluble, whilst polymerised (solid) oils contain an insoluble portion. In every case in which unchanged polymerised wood oil was used in linoleum cement it could be recognised by this method. *Resins and Chinese wood oil in cements*:—From experiments on these lines the following conclusions were drawn:—(1) At least 20% of resins must be present to obtain the maximum conversion of the oily matter into a substance with high binding capacity. (2) If rosin is the only resin present, the "body" (as measured by the linoxyn) is liable to fall below the practicable limit. (3) The presence of gum kauri is essential for the reduction of greasiness (petroleum spirit extract). (4) Chinese wood oil (the use of which has been patented as a substitute for kauri gum) has no chemical influence on the transformation, but is disadvantageous as it reduces the amount of oxidised oil in the mixture. (5) The best proportions for the three ingredients are about 80% of oil, 10% of kauri gum, and 10% of rosin. —C. A. M.

#### PATENTS.

*Drying rooms especially intended for baking enamel on to metallic surfaces.* A. E. White, London. From Reliance Drying Room Co., Chicago. U.S.A. Eng. Pat. 102,539, Feb. 9, 1916. (Appl. No. 1929 of 1916.)

The drying chamber is provided with a turn table floor, part of which (substantially a half) projects outside. This floor is divided into, preferably equal, sections by a vertical partition, which moves with the floor and in one position engages with the walls to close the chamber. The heating compartment surrounding the inner walls of the chamber is preferably made to conform sub-

stantially to the shape of the inner portion of the turntable, whilst a fan or blower may be provided at the top to deliver downward currents of hot air into the drying chamber. Whilst the charge of material on the portion of the floor within the chamber is being dried, a fresh charge is being arranged on the portion of the floor outside the chamber.—C. A. M.

*Enamel on metallic surfaces; Air-circulating systems for drying rooms for baking*—A. E. White, London. From Reliance Drying Room Co., Chicago. U.S.A. Eng. Pat. 102,885, Feb. 9, 1916. (Appl. No. 7040 of 1916.)

The drying room comprises a drying and heating chamber in communication with each other at the top and bottom of a vertical partition separating them. A pipe system communicating with the chambers at the bottom leads upward therefrom to a fan or blower which discharges air downwards into the drying chamber; ventilating pipes leading from this chamber are provided for removal of foul or saturated air; these converge together into a single flue to a suitable point of discharge. The system is preferably used in conjunction with a drying room provided with a turntable floor (see preceding abstract).—T. H. B.

*Phenols, formaldehyde, and salts of sulpho-acids; Process for obtaining solid products of condensation from*—K. Tarassoff, Moscow, and P. Sheshtakoff, Petrograd. Eng. Pat. 102,635, Jan. 20, 1916. (Appl. No. 956 of 1916.)

SOLID, infusible, and insoluble products are obtained by condensing phenols with formaldehyde in presence of ammonium, alkali, or alkaline-earth salts of sulpho-acids (preferably naphtha sulpho-acids or sulpho-aromatic fatty acids), and of an acid which does not decompose such salts. For example, 300 parts by weight of "commercial carboic acid 99—100% cresols," is gradually mixed with 135 parts of a 40% solution of sodium salts of naphtha sulpho-acids, 9 parts of hydrochloric acid (sp.gr. 1.14), and 150 parts of 40% aqueous formaldehyde, and heated to 65°—70° C. A resinous liquid mass separates with a watery layer on top; the latter is removed and the resinous mass heated until there is a loss of 70—75 parts by weight of the total materials used. After cooling to 35°—45° C., 120 parts by weight of aqueous 40% formaldehyde is added and the whole mixed to a homogeneous mobile liquid, which is poured into moulds, left to rest some hours, then gradually heated for one hour to 45° C., then, at intervals, by 10°—15° at a time, to 100° C.—T. H. B.

*Hard compositions; Production of*—from formaldehyde and phenols with carbohydrates, albuminous materials, and tannins. K. Tarassoff, Moscow, Russia. Eng. Pat. 102,751, Jan. 19, 1916. (Appl. No. 892 of 1916.)

THE process described in Eng. Pat. 7560 of 1914 (see also Eng. Pats. 528, and 6716 of 1914; this J., 1914, 557; 1915, 187, 1201), according to which organic bodies containing no aldehyde, phenol, or sulpho-groups, or substances in which these groups are not essential constituents, may be caused to take part in the reactions between phenols and formaldehyde, in the presence of one or more organic sulpho-acids, is now modified by the inclusion of carbohydrates, albuminous materials, and tannins amongst these "fillers." For example (1) carboic acid, 100; a 37% solution of naphtha sulpho-acids, 20; egg albumin, 20; and water 50 grms. are heated at 75°—80° C.



until the added water is evaporated. The mass is cooled and formaldehyde (40%), 40 grms., is added. When the reaction, which takes place at 60°–80° C. moderates, a further 50 grms. of water is driven off by heating, the mass cooled to 25°–30° C., and formaldehyde (40%) 50 grms., added. The viscous liquid is poured into moulds and left for 10–12 hrs., and the hardening process is then completed by heating up to 75° C. for half-an-hour, up to 85°–90° C. for 1–2 hours, and at 100° C. for an equal length of time; 205 grms. of a very hard, elastic, non-fusible, non-transparent, bluish-yellow solid is obtained. The egg albumin may be replaced by blood albumin, gelatin, casein, glue, etc. (2) Carbolic acid, 100; formaldehyde (40%), 50; sulpho-acid solution, 50; and wheat starch, 30 grms. are heated together to 40°–45° C.; heat is evolved and a jelly is formed which thins on boiling, the starch being deposited. The mixture is rendered homogeneous by stirring, and 55–60 grms. of water is then evaporated by heating. The mass is cooled to 20°–25° C., formaldehyde (40%), 40 grms., is added, and the viscous tarry mass is then poured into moulds and heat-treated as in example (1); 200–215 grms. of hard, rose-coloured, infusible, elastic material is obtained. (3) With carbolic acid, 100; sulpho-acid solution (40%), 50; crystalline tannin solution (50%), 60; and formaldehyde (40%), 40 grms., heated to 60°–80° C., and then until 70–75 grms. of water has been driven off, subsequently cooled, mixed with formaldehyde (40%) 50 grms., and hardened off in moulds as before, a hard, infusible, "chestnut-yellow" mass is obtained.—E. W. L.

*Vulcanised oils; Preparation of solutions of—*  
obtained by the action of sulphur chloride, in amyl acetate. M. Poto. Fr. Pat. 480,939, Feb. 12, 1916.

The oil, e.g., castor oil, 1 kilo., is intimately mixed with amyl acetate, 2 litres, and sulphur chloride, 250 grms., is then stirred in. A stiff, almost colourless jelly is formed, which gives off large quantities of hydrogen chloride, resulting from the decomposition of some acetyl chloride produced during the reaction. When left in a hermetically closed vessel for several days, this product completely liquefies, the vulcanised oil passing into solution. Sodium or barium or other carbonate, or sodium theiosulphate—but preferably barium carbonate—is added to neutralise the free acid present, and the solution is then decanted and filtered. This solution may be used for impregnating fabrics and other materials, or it may be mixed with other solvents, and nitrocellulose, as well as with soluble colours and pigments, and used for varnishing black and coloured leathers and for a variety of other purposes.—E. W. L.

*White pigment of the lithopone type; Inalterable—*  
R. B. Llopert. Fr. Pats. 480,846 and 480,847, Feb. 2, 1916. Under Int. Conv., Feb. 19 and Mar. 16, 1915.

SEE U.S. Pats. 1,130,427 and 1,140,354 of 1915; this J., 1915, 670, 715.

*[Resinous] condensation products; Process for the manufacture of insoluble—*  
F. Pollak, Berlin. U.S. Pat. 1,211,227, Jan. 2, 1917. Date of appl., Aug. 15, 1912.

SEE Fr. Pat. 447,969 of 1912; this J., 1913, 436.

*Process of manufacturing bronze powder.* Fr. Pat. 480,504. See X.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

### PATENTS.

*Vulcanisation; Circulation and exhausting apparatus for—*  
A. E. Alexander, London. From Goodyear's Metallic Rubber Shoe Co., Naugatuck, Conn., U.S.A. Eng. Pat. 102,802, Aug. 29, 1916. (Appl. No. 12,204 of 1916.)

In apparatus for vulcanising rubber boots and similar articles, in which a difference of pressure is maintained between the inside of the perforated forms on which the articles are mounted and the heated fluid (gaseous) medium surrounding the articles, a certain leakage of the medium (e.g., carbon dioxide) into the interior of the forms occurs. Loss of heat units and of heating medium is minimised, according to the invention, by inserting an exhaust pump in series with the low pressure and the high pressure sides and thus returning the gaseous leakage quickly through a preheater, to the high pressure atmosphere of the vulcaniser. Good circulation is ensured by running a branch pipe from within the vulcaniser, opposite the gas inlet, to the suction end of the pump. The interior of the forms communicates with the hollow frame of the trolley which carries them, and this in turn is connected with the external suction pipe of the pump.—E. W. L.

*Vulcanisable composition; Process of manufacture of a—*  
J. S. Campbell. Fr. Pat. 479,960, Oct. 8, 1915.

FINELY divided leather is heated with oil or fat, and whilst continuing the heating, caustic soda or potash (sufficient to saponify the oil), vegetable fibrous material, magnesia, filling material, such as steatite or chalk, and a binding material such as gum or caoutchouc are successively added at intervals, and the whole thoroughly mixed. The mass is then dried and worked up by passing between cylinders, during which operation sulphur or some other vulcanising agent is added. The material may then be made into sheets, etc., and vulcanised.—F. C. T.

## XV.—LEATHER; BONE; HORN; GLUE.

### PATENTS.

*Skins; Deliming and bating of—*  
J. Meister. Fr. Pat. 480,196, Nov. 10, 1915.

SKINS are submitted to the action of pepsin or other suitable enzymes in very dilute hydrochloric acid, the temperature being maintained at 40° C. Other acids (boric, lactic) and also salts (ammonium chloride) are added to keep up the hydron concentration. The process is much more rapid and reliable than the usual one.—F. C. T.

*Tanning extracts; Manufacture of—*  
Soc. Anon. des Matières Tannantes et Colorantes. Fr. Pat. 480,300, Nov. 23, 1915.

AN extract suitable for tanning is prepared from the pods of *Acacia arabica* and *Acacia Senegal* or of other similar trees.—F. C. T.

*Tanning.* Gum Tragacanth Supply Co., Ltd. Fr. Pat. 480,456, Dec. 16, 1915.

SEE Eng. Pat. 7635 of 1915; this J., 1916, 748.

*Process for utilising osier-bark.* Fr. Pat. 480,637. See V.

*Manufacture of a vulcanisable composition.* Fr. Pat. 479,960. See XIV.



**XVI.—SOILS; FERTILISERS.**

*Phosphoric acid; Valuation of water-soluble as compared with citrate-soluble*——. E. J. Pranke. J. Ind. Eng. Chem., 1917, 9, 54—55.

In the reports of some of the American experimental stations the "valuations" or "trade-values" for water-soluble phosphoric acid have been put at higher figures than those for citrate-soluble phosphoric acid, although these "values" are not intended to show the agricultural value of the products. Moreover they are not justified by the selling prices. Thus the cost of the available (water-soluble plus citrate-soluble) phosphoric acid in acid phosphate (superphosphate) decreases with the rise in the proportion of water-soluble to citrate-soluble phosphoric acid, whilst citrate-soluble acid in organic phosphates costs more per unit than does the available acid in superphosphate.—C. A. M.

*Third report on the improvement of indigo in Bihar.* Howard. See IV.

*Relationship existing between oxydase activity of plant juices and their hydrogenion concentration, with a note on the cause of oxydase activity in plant tissue.* Bunzell. See XVIII.

**PATENTS.**

*Manures; Manufacture of compound*——. C. De-guise. Fr. Pat. 480,500, Dec. 30, 1915.

INSOLUBLE phosphates are transformed into soluble products by the action of sodium bisulphate, dissolved in water or in liquids containing fertilising elements in solution or suspension.—B. N.

*Manure; Soluble phosphatic—and process of manufacture.* E. Stoppani and V. Volpato. Fr. Pat. 480,697, Jan. 17, 1916. Under Int. Conv., Jan. 26, 1915.

A SOLUBLE phosphatic manure is formed by mixing natural phosphates with about 6% of carbonates, oxides, or salts of alkali or alkaline-earth metals, and then heating to about 600°C., afterwards hydrating, preferably whilst hot, by the addition of water.—B. N.

*Calcium cyanamide; Apparatus for the treatment [granulation] of*——. G. W. Sinclair. Odda, Norway. U.S. Pat. 1,211,252, Jan. 2, 1917. Date of appl., July 7, 1914.

SEE Eng. Pat. 9821 of 1914; this J., 1915, 916.

*Peat [as fertiliser]; Utilisation of*——. Wetcar-bonizing, Ltd. Fr. Pat. 480,079, Oct. 26, 1915.

SEE Eng. Pat. 18,838 of 1914; this J., 1916, 133.

*Phosphates; Manufacture of citrate-soluble*——. S. B. Newberry. Fr. Pat. 480,355, Nov. 30, 1915.

SEE U.S. Pats. 1,162,802 and 1,162,044 of 1915; this J., 1916, 133.

*Manufacture of cellulose, and utilisation of the waste lyes.* Fr. Pat. 477,895. See V.

**XVII.—SUGARS; STARCHES; GUMS.**

*Invertase; Presence of*—— in cane sugars from Demerara and St. Domingo. H. Pellet. Bull. Assoc. Chim. Sucr., 1916, 33, 263—266.

MAQUENNE has detected the presence of invertase in raw cane sugars from Demerara and St.

Domingo, particularly the former. It originates, doubtless, from micro-organisms, probably bacteria of the type described by Lewton-Brain and Deerr (this J., 1900, 845), which develop in the finished sugar under suitable conditions of humidity and temperature. The presence of invertase explains why in order to avoid rapid decomposition of sucrose, liquors should be submitted as soon as possible to a temperature of 90°—100°C., and also why the presence of certain antiseptics is not efficacious in preventing alteration of the syrups and other products. It suggests that to obviate decomposition of solutions of cane sugar products, it is desirable to destroy the enzyme by heating to a moderate temperature in presence of an alkali, and also to operate with highly concentrated solutions.—J. P. O.

*Clarification [of sugar cane juices]; Processes for the*—— with direct filtration through presses. C. Friboing. Bull. Assoc. Chim. Sucr., 1916, 33, 227—255.

THE author has shown previously that after treatment with kieselguhr and dicalcium phosphate, it is possible to filter defecated cane juice in its entirety through filter-presses and obtain a clear liquor and good cake, instead of following the present more lengthy system of decantation and after-treatment of the "bottoms" and scums. Further details are now given, and it is pointed out that the total filtration of juice in this way is impossible when soluble phosphates alone (that is, phosphoric acid and monocalcium phosphate from concentrated superphosphate and acid phosphate) are added to the juice already sulphited and limed as usual, the effect being to produce a gelatinous precipitate which prevents filtration in presses. On the other hand, kieselguhr alone at the rate of 2.5 kilos. per 1000 litres of juice produces the desired effect, although no appreciable increase of purity results. Soluble phosphates in conjunction with kieselguhr cause an increase of purity, while allowing of direct filtration, but a relatively large amount of kieselguhr is required. Economical and generally satisfactory results with an appreciable increase of purity are stated to be obtained by the use of a mixture of kieselguhr and dicalcium phosphate (insoluble) in the proportion of 2:1, and employing 3—4 kilos. (or 4—5 kilos. with low purities) per 1000 litres of the juice after it has been submitted to sulphiting and liming as usual. Under these conditions the filtering surface required will be 50 sq.m. per 100 tons of cane worked, the process is rapid and simple, and there is a marked saving in labour and steam. The amount of press-cake (dry state) obtained per 1000 litres of juice, with ordinary sulphitation and defecation, is 6—8 kilos., while with the further addition of both kieselguhr and dicalcium phosphate it is 11—12½ kilos., and all or almost all the phosphoric acid added can be recovered with that normally present in the juice, giving a cake containing 5 to 14% of phosphoric acid, 7 to 8% of nitrogen, and 10 to 20% of lime, which forms a useful fertiliser in the cane field.—J. P. O.

*Sugar; Quantitative determination of minute amounts of*—— by  $\alpha$ -naphthol and the estimation of entrainment losses [in sugar factories]. A. F. Blake. Int. Sugar J., 1917, 19, 26—28.

TRACES of sucrose in water, up to 100 parts per million, can be estimated, with a possible error of 20% for pure solutions, by the following method. Ten c.c. of concentrated sulphuric acid is added carefully to 5 c.c. of the liquid under test containing 5 drops of a 20% solution of  $\alpha$ -naphthol in pure alcohol; the mixture is stirred vigorously and after 2 mins. the purple colour

produced when sugar is present is compared with a series of standards. The standards for concentrations of sugar up to 20 parts per million are made by mixing solutions of cobalt nitrate and copper sulphate; those for larger amounts are made from red and blue organic dyestuffs and require to be renewed about once a month. Vessels of uniform size should be employed and should not be wiped with a cloth before use. The presence of iron in the liquid interferes somewhat with the test, and products from the destructive distillation of low-grade materials vitiate it entirely. With the salt water from condensers the test has given perfect satisfaction. The author applies it to the examination of condensed water from evaporation, employing a device illustrated in the original paper) for taking periodic samples from the condenser fall pipe; by this means the losses from evaporator entrainment are determined regularly.—J. H. L.

*Sucrose; Simplified inversion process for the determination of — by double polarisation.* H. S. Walker. Hawaiian Chem. Assoc., Oct. 12, 1916. Int. Sugar J., 1917, 19, 31—35.

The following method of inversion, applied to the determination of sucrose in pure sugar and in waste molasses, gave percentage values rarely differing by more than 0.1 from those obtained by Herzfeld's method. Seventy-five c.c. of the solution to be inverted (normal strength for sugars and half-normal for molasses), in a 100 c.c. flask, is heated in a water bath to 65° C., then withdrawn from the bath, treated with 10 c.c. of a mixture of equal volumes of hydrochloric acid (sp. gr. 1.183) and water, and allowed to cool spontaneously in the air for 15 mins. or as much longer as may be convenient. After being cooled to the ordinary temperature the liquid is made up to 100 c.c. and polarised as usual. In the case of products which have been clarified with a large excess of basic lead acetate, the excess alkalinity is neutralised before heating, e.g., by addition of 1 or if necessary 2 c.c. of the dilute acid. A considerable latitude appears to be permissible as regards the temperature of the sugar solutions when the acid is added (63°—70° C.), and the rate of cooling as influenced by the temperature of the laboratory (in the author's experiments this seems to have been in the neighbourhood of 26° C.) and the thickness of the glass flasks.—J. H. L.

*Sugar; Report on —.* [Modification of the Clerget process of double polarisation. Use of invertase as hydrolyst in the double polarisation method.] C. A. Browne. J. Assoc. Off. Agric. Chem., 1916, 2, 134—143.

In the original Clerget process, in which 50 c.c. of the normal or half-normal sugar weight solution is made up to 55 c.c. with hydrochloric acid and inverted, there is a diminution in volume due to the following three causes: (1) the contraction following on inversion; (2) the increase of temperature produced by the addition of the hydrochloric acid; and (3) the evaporation of water from the neck of the flask during heating, the combined influence of these causing the diminution of the volume of the 55 c.c. by 0.33 c.c. for the half-normal weight of 13 grms. In order to eliminate all three sources of error, it is proposed to make up the volume of the solution after and not before inversion, the process being thus made more exact than any modification involving dilution to 100 c.c., in which any error is multiplied by 2. It is further recommended that inversion be made at the ordinary temperature over-night. The sucrose is calculated by the following formula allowing for differences in specific rota-

tion caused by varying concentration when less than the normal weight of 26 grms. is taken; A is the direct polarisation and B the invert polarisation corrected for increase of volume, and t is the temperature at which the invert solution is read:—

$$144.9 - \frac{t}{2} - 0.01 \left[ 144.9 - \frac{t}{2} - (A - B) \right]$$

Hudson's method of double polarisation using invertase as hydrolyst (this J., 1910, 443), in which the lead used for the clarification of the product is eliminated by sodium carbonate or potassium oxalate, is shown to give far more accurate results than the method of acid inversion in the analysis of complex mixtures containing sucrose and levulose. For purposes of commercial analysis, however, it cannot at present displace acid inversion, the preparation of the invertase solution being troublesome, and the whole process requiring much care and watchfulness, but it is invaluable as a control upon other methods.—J. P. O.

*Decolorising carbons; Action of different — on pure and impure sugar solutions, and their influence in the analysis of sugar factory products.* H. Pellet. Bull. Assoc. Chim. Sucr., 1916, 33, 220—227.

DECOLORISING carbons of different origin vary considerably in their properties. Thus, "Eponit" (this J., 1911, 146, 1177) has a density of 250 grms. per litre, carbon from the shells of cherry-stones one of 573, while ordinary unwashed animal charcoal has one of 895. Similarly, their decolorising power and ash content show wide differences, the latter varying from 1.5% (cherry-stone carbon) to 81.8 (animal charcoal) per 100 of dry substance. When used as clarifying agents in the double polarisation method of determining sucrose, all adsorb a greater or less quantity of sugar, and with 100 c.c. of a pure solution of sucrose, originally polarising 20.85°, the reading after the addition of 2 grms. of the different grades was as follows: "Eponit," 25.80°, animal charcoal, washed successively with hydrochloric acid and water, and dried, 26.20°, ordinary unwashed animal charcoal, 26.20°, and cherry-stone carbon, 26.80°. In the case of solutions of sugar factory products defecated with basic or normal lead acetate, the adsorption is less, while in that of liquids first treated with basic lead acetate and afterwards with excess of sulphurous acid, that is under the conditions of the sulphurous acid direct polarisation method, practically no diminution could be observed. On the contrary, a slight increase, averaging 0.15° with a polarisation of about 28.0°, was noticed, this being apparently due to the adsorption of lead salt, the effect of which on the optically-active amino-acid derivatives causing a levorotation was thus modified. An adsorption of optically-active substances in unf fermented cane vinasse by decolorising carbons was also demonstrated, but cupric reducing substances contained in both beet and cane molasses do not appear to be affected.—J. P. O.

*Raffinose; Determination of — in presence of sucrose by double inversion using top and bottom fermentation yeasts.* H. Pellet. Bull. Assoc. Chim. Sucr., 1916, 33, 255—262.

Owing to the presence in beet sugar factory products of optically-active amino-acids and their derivatives, the rotation of which depends upon the reaction of the solution, whether alkaline or acid, the ordinary method of determining raffinose by acid inversion is subject to an appreciable error. On examining the method of double fermentation by top and bottom yeasts suggested by Bau, and elaborated by Hudson and Harding (this J., 1915,

1065), the author obtained good results, both in pure solutions as well as in the presence of dextrose, levulose, lactose, maltose, and other carbohydrates. In the case, however, of very impure liquids, as molasses, though equally reliable results are obtainable, fermentation is very slow, particularly with the bottom fermentation yeast, and demands about 7 hours, sometimes much longer. It is important that both the fermented liquids should be examined at the same temperature, and in order to avoid any error from adopting corrections, it is advisable to read throughout at 20° C. With a product obtained by the desaccharification of molasses by the strontia process, it was observed that the difference between the ordinary and the acid direct polarisations was very slight, this being due to the fact that the optically-active substances capable of causing differences are almost entirely removed with the mother-liquors, after their decomposition.—J. P. O.

*Carbohydrates; Methods for the estimation of mixtures of four or more—involving oxidation with bromine.* E. G. Wilson and W. R. G. Atkins. *Biochem. J.*, 1916, 10, 504—521. *Analyst*, 1917, 42, 19—20.

DEXTROSE and maltose are quantitatively oxidised by bromine at the ordinary temperature when their solutions are saturated with bromine and allowed to stand; the liquid, however, still retains a small amount of its original cupric-reducing power. Levulose is not oxidised at all, or only to a very small extent, under these conditions, whilst in presence of N/10 sulphuric acid there is a slight loss of levulose in 48 hours at the ordinary temperature. A mixture of sucrose, maltose, dextrose, and levulose may be analysed as follows:—The sucrose is determined by polarisation and reduction before and after treatment with invertase. The resulting mixture of reducing sugars is treated with bromine under standard conditions of acidity and temperature for a given time (see following abstract), whereby dextrose and maltose are oxidised, whilst levulose remains unaffected: the reducing power of the solution may be taken as due to levulose only, the result being corrected for the portion derived from the sucrose. By subtracting the rotation due to sucrose and levulose from the initial rotation, the rotation due to dextrose and maltose is obtained, and in like manner by subtracting the reduction due to levulose from the initial reduction, the reduction due to dextrose and maltose is obtained. The amounts of dextrose and maltose can then be calculated.

It was observed that addition of dilute alkali (N/2 or 2N sodium hydroxide) to a solution of dextrose, levulose, or maltose, produces a large temporary increase in the reducing power of the solution.

*Honey and other substances containing levulose; Analysis of —.* W. R. G. Atkins. *Analyst*, 1917, 42, 12—13.

THE method of Wilson and Atkins (see preceding abstract) of oxidising sugars other than levulose by means of bromine is rapid, and although less accurate than the fermentation method of Davis and Daish (this J., 1913, 1025), gives fairly good results in the analysis of mixtures of dextrose, levulose, and maltose. An excess of bromine is added to the solution, which should contain sufficient sulphuric acid to make the strength decinormal, and the flask is kept in constant agitation for 42 hours at the ordinary temperature. After the oxidation the maltose retains only a trace of its cupric-reducing power and the dextrose about 1% thereof, whilst the levulose shows a reduction of approximately 1 to 2% in reducing power

owing to its decomposition by the N/10 sulphuric acid and the hydrobromic acid produced during the oxidation of the other sugars. This loss in the quantity of levulose is approximately balanced by the residual reducing power from the other sugars. After removal of the excess of bromine by means of gaseous sulphur dioxide and finally by titration with a solution of sulphurous acid, the solution is rendered only slightly acid or neutral (but not alkaline) by the addition of potassium carbonate, and the reducing power of an aliquot portion is determined. For this purpose Kendall's solution (this J., 1912, 351) is suitable as it does not dissolve the cupric oxide, and the amount of levulose may then be found by reference to the author's table (*Biochem. J.*, 1916, 10, 137). In the case of honey or other mixtures containing only dextrose and levulose, sufficiently accurate results are obtained by determining the reducing power before and after the oxidation. For the determination of sucrose in honey the reducing power is determined before and after inversion, and the inverted solution afterwards oxidised with bromine, due allowance being made for the dextrose and levulose produced by the inversion. The method of oxidation with bromine affords an easy means of detecting abnormal quantities of dextrose in honey, jams, etc.—C. A. M.

*Fructose [levulose]; Evidence indicating the existence of a new variety of —. A reactive form of methylfructoside.* J. C. Irvine and G. Robertson. *Chem. Soc. Trans.*, 1916, 109, 1305—1314.

IN a previous paper (this J., 1915, 627) some derivatives of a new highly reactive form of glucose (dextrose),  $\gamma$ -glucose, differing from the well-known  $\alpha$ - and  $\beta$ -modifications presumably in the position of the oxidic link, were described. It would now appear that this  $\gamma$ -type is represented also in the case of the other monohexoses, galactose and fructose. One of the characters of this series is the ready formation and equally ready hydrolysis of the methylglucosides and fructosides, which are hydrolysed, like sucrose, on warming with highly dilute acids. There is a strong presumption in favour of the view that the fructose component of sucrose belongs to the new  $\gamma$ -type, whereas the glucose component is of the ordinary type. The evidence on which the view of the existence of " $\gamma$ -fructose" is based is largely contained in work by Purdie and Paul (*Chem. Soc. Trans.*, 1907, 91, 289) on the formation of tetramethylfructose from the crude methylfructoside obtained, according to Fischer, by the condensation of fructose with methyl alcohol containing 0.5% of hydrogen chloride. This reaction proceeds readily at 35° C. and in this respect shows a marked difference from the mode of preparation of alkylglucosides where prolonged heating at 100° C. is generally required. The product is obviously a mixture and its resolution is complicated by the fact, now first ascertained, that the portion of the methylfructoside corresponding with the new  $\gamma$ -modification of the sugar combines readily with whatever traces of acetone may be present in the methyl alcohol employed, yielding methylfructoside-monoacetone, which can only be isolated by fractional distillation under the highest vacua afforded by the Gaede pump. Two distinct tetramethylfructoses, a crystalline and a liquid modification, are formed from the mixture, which must contain at least four isomeric methylfructosides; two of the latter correspond to the ordinary form of fructose ( $\alpha$  and  $\beta$ ) and two to the new  $\gamma$ -form which also is capable of existing in  $\alpha$ - and  $\beta$ -modifications. The specific rotation of these new modifications of fructose when in equilibrium is low and is probably dextro in sign; the melting points of the sugars would be lower than those of the ordinary

form; they may be syrups. Taking the analogy of  $\gamma$ -glucose, it is assumed that  $\gamma$ -fructose contains the ethylene oxide ring, while the ordinary fructose contains the butylene oxide linkage.—J. F. B.

*Galactose; Determination of free and combined*——  
A. W. van der Haar. Chem. Weekblad, 1916, 13, 1204—1213.

The galactose is oxidised by means of nitric acid and the resulting mucic acid is weighed, as first suggested by Creydt (*Dissert.*, Erlangen, 1888). From 0.25 to 1 gm. is mixed with 60 c.c. of nitric acid (sp.gr. 1.15 at 15° C.) in a beaker 12 cm. high by 60 mm. in diameter, which is immersed in a boiling water bath. The contents are stirred at intervals and the heating continued until the liquid is reduced to less than 20 grms., when the mixture is cooled and made up to 20 grms. with water. After the addition of 0.5 gm. of dry mucic acid which has been purified by recrystallisation from alcohol, the liquid is left for 48 hours at 15° C., and the deposit of mucic acid is then collected on an asbestos filter in a Gooch crucible, washed with 5 c.c. of water, and dried at 100° C. until constant in weight, the weight of mucic acid added to promote the crystallisation being afterwards deducted. For the determination of galactose in glucosides or polysaccharides, from 0.25 to 1 gm. of the anhydrous substance is hydrolysed with 25 c.c. of 2 to 5% sulphuric acid, and the liquid filtered. After 24 hours, from any insoluble products. The filtrate and washings are concentrated, rendered slightly alkaline with sodium hydroxide solution, made up to 30 c.c., and treated with 30 c.c. of 50% nitric acid, and as much sucrose as corresponds to the non-sugar part of the glucoside (e.g., sapogenins). The oxidation is then effected as described above. From the weights of mucic acid obtained the corresponding quantities of galactose may be found by reference to the following tables (intermediate figures may be obtained by interpolation):—

#### I. Galactose alone.

Mucic acid.	Galactose.	Mucic acid.	Galactose.
mgms.	mgms.	mgms.	mgms.
— 4	0	366	500
+0.8	10	455	600
20	50	525	700
35	100	615	800
100	150	679	880
142	200	688	890
180	250	696	900
215	300	780	1000
296	400		

#### II. Galactose made up to 1 gm. with sucrose.

Mucic acid.	Galactose.	Mucic acid.	Galactose.
mgms.	mgms.	mgms.	mgms.
— 4	0	305	300
+2.4	10	284	400
28	50	325	450
62.5	100	360	500
100	150	440	600
133	200	475	650
140	250	555	750
165	300	780	1000

—C. A. M.

*Gum-yielding plants of Brazil.* H. Montandon. Chacanas e Quintaes, 1916, 13, 417—421. Bull. Agr. Intell., 1916, 7, 1295—1296.

SUBSTITUTES for gum arabic are obtained from the following Brazilian plants:—(1) Different species of "angico," *Enterolobium ellipticum* (*Pithecolobium*

*gummiferum*); (2) "Arvore da gomma" or "gomma lagrima" (*Vochisia gummifera*), which is common in the province of Rio de Janeiro; and (3) "Vinheiro do Campo" or "Arvore do vinho" (*Vochisia thyrsoidea*), which is common in the State of Minas Geraes. The best quality or "gomma lagrima" is free from impurities and is perfectly colourless and transparent. It is completely soluble in 11 parts of cold water, yielding a colourless, transparent mucilage. A sample with sp.gr. 1.604 at 26° C. had the following composition:—Water, 11.79; arabin, 87.67; yellow bitter substance, 0.03; resinous substance 0.04; insoluble matter, 0.012; and ash, 0.44%. Experiments made by Pizarro showed that the gum of *Vochisia thyrsoidea* has 10 times the adhesive power of gum arabic, and is quite equal to it in medicinal properties. Consignments of Brazil gum have been exported to Liverpool and to Germany from 1900 onwards.—C. A. M.

*Utilisation of diseased potatoes as fodder and industrial raw material.* Wehmer. See XIXA.

#### PATENTS.

*Fondant [sugar cream]; Manufacture of*——  
P. H. Schlueter, Chicago, Ill. U.S. Pat. 1,201,357, Oct. 17, 1916. Date of appl., Mar. 8, 1916.

For making fondant or sugar-cream for filling sweetmeats, a suitable charge of sugar, e.g., 25 lb. of pure cane sugar and 5 lb. of glucose, is mixed with only sufficient water to effect complete solution on boiling, and the solution is boiled in the open air until it attains a temperature corresponding to 228° F. (100° C.) at sea level. The charge is then subjected to a vacuum as complete as possible (28—28.5 ins.) and as soon as vigorous ebullition has set in, the application of heat is discontinued and evaporation allowed to proceed under vacuum until the syrup reaches a density corresponding to a boiling point of 238° F. (114.5° C.) in the open air. During the evaporation of the syrup without the application of heat, the temperature will have become so reduced that the operation of beating to cream may proceed immediately. The apparatus may consist of a pan and gas-heated furnace mounted on lifting gear beneath a hood connected with a vacuum pump, so that boiling in the open or *in vacuo* may be carried out by lowering or raising the pan.  
—J. F. B.

*Sugar; Process for the manufacture of*——  
O. Carr, Asheville, N.C. U.S. Pat. 1,201,986, Oct. 17, 1916. Date of appl., Sept. 9, 1914.

SUGAR juice, either raw or treated with precipitating agents, is atomised into an air-chamber in which the moisture of the juice is absorbed by the air at a moderate temperature and the solids are rapidly deposited as a dry powder. The non-sugar impurities of the juice in the dried state are soluble with difficulty in cold water and especially after treatment of the original juice with lime, aluminium hydroxide, or metallic salt precipitating agents. The sugar solids are dissolved by lixiviation preferably in the cold; suitable substances may be added to the lixiviating water to prevent solution of impurities, e.g., lime, alcohol, etc. The solution may be clarified by carbonation or decolorising agents, and evaporated either by atomising or boiling to "grain" in the usual manner.—J. F. B.

*Sugar beets; Process of treating*——, and the product derived therefrom. G. H. Benjamin, New York. U.S. Pat. 1,207,840, Dec. 12, 1916. Date of appl., Dec. 17, 1914.

BET slices are dried, e.g., by air at about 170° F.

(77° C.) until about 90% of their moisture has been removed, in such a way as to coagulate the coagulable matters without destroying the enzymes, rupturing the cell walls, or rendering them incapable of permitting osmosis when the dried product is subsequently treated by the diffusion process. The dried product may be treated with a material which will prevent fermentation or mouldiness.—J. H. L.

*Bleaching liquids [sugar juices]; Apparatus for* — L. J. Foret, Lockport, La. U.S. Pat. 1,208,584, Dec. 12, 1916. Date of appl., Jan. 22, 1916.

The apparatus comprises a vertical cylinder provided with a series of annular ribs fixed to the interior wall, a series of annular shelves loosely mounted on the ribs, each formed of two semi-circular segments, a vertical rotary spindle projecting downwards through the centre of the casing and provided with a series of circular discs overlapping the inner edges of the annular shelves, and means for introducing liquid into the upper part and gas into the lower part of the casing and for discharging waste gas from the upper part and the treated liquid from the lower part.—J. H. L.

*Starch; Process for making soluble* — J. Kantorowicz, Breslau, Germany. U.S. Pat. 1,207,177, Dec. 5, 1916. Date of appl., Oct. 19, 1914.

SOLUBLE starch, solutions of which do not solidify or gelatinise at ordinary temperatures, is made by acting on starch with organic acids together with substances which convert starch into soluble starch, e.g., persalts, with or without mineral acids.—J. H. L.

## XVIII.—FERMENTATION INDUSTRIES.

*Malts; Comparative studies of floor-, drum-, and box* — L. Adler. Z. ges. Brauw., 1916, 39, 201—204, 217—221, 225—226.

THREE malts were prepared from the same barley by germinating (1) on floors, (2) in a Galland malting drum, and (3) by the Kropff system (this J., 1912, 39). All the malts were kilned under similar conditions. In germinating by the Kropff system, the barley, after four days on the malting floors, was transferred to a box in which it formed a layer 3 feet deep on a perforated floor, and in which it was aerated for about an hour after successive resting periods of 6, 6, 6, 10, 10, 24, and 24 hours. The three malts were subjected to a very thorough investigation. In almost all respects the characters of the floor malt were intermediate between those of the other two. The Kropff malt, owing to restricted respiration during the later stages of germination, showed a smaller malting loss than the others, but it was darker in colour. It was further distinguished by the activity of its diastatic, proteolytic, and acid-forming enzymes and its phosphatases, and by rapid saccharification and high yield of extract. The wort from it filtered rapidly and well, possessed a high acidity as determined by titration, and exhibited a coarsely flocculent "break." The drum-malt, on the other hand, was characterised by high malting loss, defective modification, and weak enzymic activity. It saccharified less rapidly than the others and gave a lower yield of extract. The wort from it possessed a lower acidity as determined by titration, but a higher concentration of hydrogen ions, than either of the others; this is attributed to its relative poverty in respect of proteolytic

products, which exert a "buffer" action towards acids. The quality of the drum-malt could probably have been improved by rotating the drum less frequently and reducing the aeration during the germinating process.—J. H. L.

*Fermentation; Loss [of sugar] in alcoholic* — L. Lindet. Comptes rend., 1917, 164, 58—61.

THE author designates as "fermentation loss" the proportion of sugar decomposed which cannot be accounted for, in accordance with Gay Lussac's equation, by the quantity of alcohol produced. This loss is due to the formation of various by-products accompanying yeast growth, and its magnitude in relation to the amount of yeast produced varies according to the nature of the nutritive substances present. In fermentation experiments with pure sucrose solutions containing only salts, including ammonium sulphate as source of nitrogen, the fermentation loss was on the average about 17 times as great as the weight of the yeast crop (dry substance) which itself amounted to only about 0.85% of the weight of sugar decomposed, and the loss consisted to the extent of  $\frac{1}{4}$  of carbon dioxide. The loss was practically the same when ammonium tartrate, lactate, or acetate was used instead of sulphate, and it was not much less when urea or asparagine was employed. In similar solutions, however, containing also 2% of substances such as gum arabic, tannin, or peat humus, fermentation was much more rapid, the yeast crop was approximately trebled, and the fermentation loss, relatively to the yeast crop, was reduced to one-third of its former value. These results were not very different from those obtained in media, such as beer wort, containing ready-formed proteins. The author concludes that of all the carbohydrates studied, except arabinose and xylose, sucrose is the least readily assimilated by yeast, the latter having great difficulty in synthesising proteins from sucrose and ammoniacal or amide nitrogen. On the other hand, in presence of gum arabic, tannin, etc. and ammoniacal or amide nitrogen, yeast develops almost as readily as in media containing proteins.—J. H. L.

*Beer; Pre-mashing and protein-haze. Concentration of hydrogen ions in — and in the course of its manufacture.* F. Emslander. Woch. Brau., 1916, 33, 160—171.

THE author's experience with pre-mashing confirms the statement of Windisch (this J., 1916, 1170) that this practice increases the yield of extract from malt, but using a hard brewing water he found that pre-mashing tended to render the beer more difficult to clarify. This difficulty was in large measure overcome by boiling the water before use. An explanation of the influence of pre-mashing on clarification is suggested. In an electric field the constituents of beer which are liable to separate on chilling, migrate towards the anode and are therefore alkaline in character. The author suggests that they consist of compounds of lime or magnesia with proteolytic products which, owing to the severance of peptide linkages and consequent liberation of acid groups, have acquired the power of combining with bases. Since pre-mashing assists proteolysis it is therefore likely to accentuate the formation of protein-haze in beers made from waters rich in lime and magnesia.—J. H. L.

*Beer; Sterilisation of — by filtration.* E. Zahn. Brewers' J., 1917, 53, 37—40.

THE sterilisation of beer by pasteurisation injuriously affects the flavour and character; it also arrests or

destroys the action of the enzymes, so that much of the value of the beer as a foodstuff is lost. The ordinary pulp beer filters do not sterilise effectively, not because the pulp fibres are incapable of holding back micro-organisms, but because of the nobility of the material under fluctuations of pressure; rigid filtering beds are absolutely necessary. These are constructed of kieselguhr compressed in a moist condition into blocks 12 ins. long and 3 ins. square, which are then fired in a kiln like porcelain. The blocks are turned to cylinders of 2 ins. diam., bored, and fitted with a lead-piece screwed and cemented into the bore and provided with a tubular neck. These filter cylinders are tested for efficiency by immersing them in water and subjecting them to a gas pressure of 6 lb. per sq. in., it having been determined that resistance to this pressure of gas in water corresponds with an effective resistance to the passage of micro-organisms. The filters comprise a pair of units each containing 55 filter cylinders, the beer is pumped through a pressure-governor into the inlet chambers of the units and passes through the filter cylinders into the outlet chambers and thence to the bottling machine. The governor varies the pressure in the inlet chamber as the demand for beer at the bottling machine increases or decreases the difference in pressure required to force the beer through the filters. The necks of the filter cylinders pass through orifices in two partitions forming an open chamber, so that in the event of any leakage in the fittings, the escaping beer falls into this chamber and not into the discharge chamber. The whole apparatus is sterilised with hot water at 180° F. (82° C.) before use and then tested collectively by displacing the water in the inlet chamber with carbon dioxide under a pressure of 6 lb.; if there is a defective tube bubbles of gas will be seen rising through the water and the cover must be removed and the tube plugged. A filter with 110 cylinders has a capacity of 50 pint bottles per hour. Sterilisation by filtration involves effective sterilisation of the bottles. This is done with hot alkaline solution and water in an automatic machine fitted with brushes. The bottles are cooled gradually with water sprays and pass, without handling, to the beer bottling machine.—J. F. B.

*Ammonia [from nitrogen in brewing materials]; Determination of—by the boric acid method.* L. Adler. *Z. ges. Brauw.*, 1910, 39, 161—164, 169—172. (See Winkler, this J., 1913, 483; 1915, 79.)

In applying Winkler's method to the determination of nitrogen in brewing materials, the author obtained accurate results by using a water-cooled condenser dipping (for the first 15 mins. of distillation) into 50 c.c. of a 4% solution of crystallised boric acid, this quantity of acid sufficing for the quantitative absorption of amounts of ammonia up to 70 mgrms. and possibly beyond. The distillate after addition of a few drops of a 0.05% solution of methyl orange was titrated with standard sulphuric acid with the help of a colour standard prepared by treating 250 c.c. of distilled water with a few drops of methyl orange and 0.15 c.c. of N/10 sulphuric acid.—J. H. L.

*Rum distillery; Scientific control of a—* F. I. Seard. *Int. Sugar J.*, 1916, 18, 498—502.

For the complete control of a rum distillery, the data required are as follows: (1) the amount of "sweets" (as dextrose) entering into the process; (2) the degree of attenuation, from which the amount of alcohol formed by fermentation is calculated and compared with the theoretical quantity to be expected from the "sweets" used to set up the wash; (3) the amount of spirit

actually distilled, and the relation between it and the theoretical amount; (4) the examination of the spent wash for alcohol as a check upon the operation of the still; and (5) the amount of esters in the spirit. In addition the obscuration must be determined, i.e., the error in the determination of the alcohol concentration by the hydrometer due to the colouring matter added to the rum. "Faults" are indicated by diluting the rum with twice or its own volume of water and allowing it to stand in a cylinder for 24 hours, any cloudiness formed being due to: (A) resinous matter from the wood of the cask; (B) the use of a too highly burnt colouring; or (C) the presence of substances of the fusel oil class, which during distillation should have been retained in the low wines.—J. P. O.

*Oxydase activity of plant juices and their hydrogen ion concentrations; Relationship existing between—, with a note on the cause of oxydase activity in plant tissue.* H. H. Bunzeli. *J. Biol. Chem.*, 1916, 28, 315—333.

WHILST the results of the experiments recorded do not establish clearly the quantitative relationship existing between the extent of the oxydase activity of a plant material and the hydrogen ion concentration existing in the medium, it appears that this relationship consists of two factors—one the direct destructive effect of the acidity on the active matter, the other the retardation of the rate of oxidation by the hydrogen ions. The hydrogen ion concentrations corresponding with complete inhibition cover a rather narrow range for each type of plant material (for potato, 3.55 to 3.70; for tulip tree material, 2.30 to 2.80), and it would seem that this acid sensitiveness is nearly constant for different genera of the same family. It would appear that the oxydase activity of plants is controlled by some constituent which is easily destroyed by acids, by heat, or by alcohol, and which changes gradually to an inactive form on standing in aqueous solution, but will keep practically indefinitely when dry. Proteins would fulfil these requirements; like all colloids, the proteins in tissues may exist in a greater or less degree of dispersion and have a more or less active surface accordingly. Condensation of oxygen on the surface of the colloid particles may explain the oxydase activity either by means of the greatly increased rate of reaction due to increased oxygen concentration, or by an increased oxidation potential of the oxygen when in such condition.—W. P. S.

#### PATENTS.

*Grape juice and process of preparing the same.* A. Fonyo. Chicago, Ill. U.S. Pat. 1,207,014, Dec. 5, 1916. Date of appl., Mar. 25, 1916.

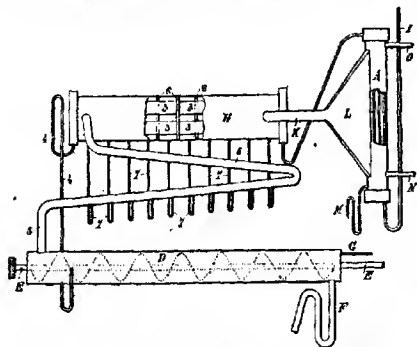
THE acid-content of grape juice is increased by a proportion of a palatable acid, e.g., 0.1—1.0% of lactic acid, which may be produced by lactic fermentation.—J. H. L.

*Distillation; Apparatus for continuous—* E. A. Chenard. Fr. Pat. 480,558, Dec. 24, 1915.

THE liquid to be distilled, e.g., alcohol, passes by the pipe, 1, to the tubular heater, A, where it is raised to a temperature below 75° C., and thence through the tubes, 3, and pipe, 4, to the cylinder, D. A rotating shaft, E, carries a helical conveyor which transports the liquid to the outlet, F. Steam entering by the pipe, G, takes up vapour from the heated liquid and carries it through the pipe, 5, to the cylinder, H, provided with baffles, 6. Progressive condensation occurs by contact with the



tubes, 3, containing cooler liquid, and the condensed liquid passes by pipes, 7, to the vapour pipe, 5, so that the vapour becomes richer in alcohol as it passes to the outlet, K. An enlarged



mouthpiece, L, admits the vapour to the preliminary heater, A, and the mixture is fractionated by withdrawing separate portions from the pipes, O, N, and condensed liquid from the pipe, M.  
—W. F. F.

*Alcohol of high strength and strong aroma; Process of manufacturing — particularly colonial rums.* P. M. H. Hervé. Fr. Pat. 480,601, Dec. 10, 1915.

THE wash is distilled through any ordinary distillation column so as to produce a distillate containing about 25% of alcohol; the distillate is then re-distilled through a rectifying column to obtain a spirit containing 77% of alcohol.  
—W. P. S.

*Industrial preparation of nitred cream of tartar [potassium and sodium bitartrates]; and soda cream of tartar [sodium bitartrate].* Fr. Pat. 480,620. See VII.

## XIXA.—FOODS.

*Phosphatides in milk; Distribution of —.* T. B. Osborne and A. J. Wakeman. J. Biol. Chem., 1916, 28, 1—9.

ALCOHOL removes from milk casein, which has been precipitated by dilute hydrochloric acid, about the same quantity of phosphatides as was obtained previously from the lactalbumin separated by heating the filtrate from the casein (this J., 1915, 883); since the amount of casein is more than six times that of the lactalbumin, the proportion of phosphatides in the casein is correspondingly less. When skimmed milk, freed from casein and heat-coagulable proteins, is neutralised with sodium hydroxide, the precipitate formed contains a very small amount of the same phosphatides and fatty substances that can be obtained from the alcohol washings of the lactalbumin; the non-protein fractions of fat-free milk contain only minute traces of phosphatides. The total quantity of phosphatides present in 1 litre of whole milk is about 27 mgrms. Phosphatides are intimately associated with the protein constituents of milk and are possibly combined with them as "lecithaluminins." —W. P. S.

*Casein; Influence of carbohydrates on the accuracy of the Van Slyke method in the hydrolysis of —.* E. B. Hart and B. Sure. J. Biol. Chem., 1916, 28, 241—249.

THE presence of carbohydrates during the hydrolysis

of casein by the Van Slyke method brings about a total redistribution of the amino-acids and this varies with the nature of the carbohydrate. The redistribution is particularly marked in the hexone bases and a decided loss of amino-nitrogen also takes place when the protein is hydrolysed in the presence of xylan. The method of direct hydrolysis is, therefore, unreliable when applied to the determination of amino-acids in feeding stuffs; owing to the variation in the nature and quantity of the carbohydrates in feeding stuffs, it is impossible to establish factors of correction for the results. (See also Gortner, this J., 1916, 1030; Roxas, *ibid*, 1916, 1174.) —W. P. S.

*Kafrin, an alcohol-soluble protein from kafr (Andropogon sorghum).* C. O. Johns and J. P. Brewster. J. Biol. Chem., 1916, 28, 59—65.

A NEW alcohol-soluble protein was separated from kafr seeds; it constitutes more than one-half of the protein in the seed, and has been named *kafrin*. It resembles zein in its composition, but differs from the latter in that it coagulates readily. Kafrin contains tryptophane and lysine, both of which are lacking in zein, and, when examined by the Van Slyke method, differences are found in the distribution of nitrogen in the two substances. Kafr is now an important cereal crop in the United States. In 1910, three million acres was under cultivation and the value of the crop was \$30,000,000. —W. P. S.

*Proteins from the Jack bean (Canavalia ensiformis).* D. B. Jones and C. O. Johns. J. Biol. Chem., 1916, 28, 67—75.

Two globulins, canavalin and concanavalin, and an albumin of the legumin type were isolated from the jack bean. The distribution of nitrogen in canavalin and the albumin was:—

	Canavalin.	Albumin.
	%	%
Humic nitrogen .....	0.28	0.23
Amide nitrogen .....	1.41	1.16
Basic nitrogen .....	8.17	8.73
Non-basic nitrogen .....	11.55	11.18
Total nitrogen .....	16.41	16.30

—W. P. S.

*Proteins of the peanut, Arachis hypogaea. The globulins arachin and conarachin.* C. O. Johns and D. B. Jones. J. Biol. Chem., 1916, 28, 77—87.

Two globulins, arachin and conarachin, separated from peanut (earthnut) meal, contained 0.4 and 1.09% of sulphur, respectively. Arachin contained 4.96%, and conarachin 6.55% of basic nitrogen, the latter being the highest percentage of basic nitrogen recorded for any seed protein. Peanut cake will probably prove highly effective in supplementing food products made from cereals and other seeds the proteins of which are deficient in basic amino-acids. —W. P. S.

*Protein-copper compounds.* T. B. Osborne and C. S. Leavenworth. J. Biol. Chem., 1916, 28, 109—123.

WHEN copper sulphate solution was added to a slightly alkaline edestin solution, the whole of the protein was precipitated when the mixture became neutral; further addition of copper sulphate resulted in the formation of a basic salt; by adding sodium hydroxide solution to a mixture of edestin and copper sulphate solution, edestin free from



copper was at first precipitated, whilst with further addition of alkali the basic copper salt was precipitated. It was possible to obtain an edestin-copper compound containing 35% of copper, but this compound was unstable. A compound which gave the biuret coloration in alkaline solution contained 12.7% of copper. Similar results were obtained in the case of gliadin. The precipitates did not represent definite copper compounds of the proteins, the amount of copper hydroxide available for combination being determined by the quantity of sodium hydroxide present in the protein solution.—W. P. S.

*Rices of Bihar and Orissa.* J. N. Sen. Arg. Research Inst., Pusa, Bull. No. 62, 1916, pp. 1-20.

EIGHTEEN samples of rice grown on Government farms in Bihar and Orissa, India, were examined; the average results obtained were as follows:—

	Husked rice.	Polished rice.
	%	%
Moisture.....	11.95	16.87
Ether-extract .....	2.36	0.88
Proteins.....	7.48	7.25
Soluble carbohydrates ..	75.86	79.99
Woody fibre .....	0.76	0.29
Ash.....	1.69	0.79
Phosphoric acid .....	0.70	0.36
Potash.....	0.36	0.22

"Husked" rice indicates rice which was simply husked, i.e., rice "in the bran," whilst "polished" rice had had much of the "skin" and germ removed by the polishing operation. The composition of the rices is similar to that of other rices, especially of Burma rice.—W. P. S.

*Potatoes; Utilisation of diseased — as fodder and industrial raw material.* C. Wehmer. Jahresversamml. Ver. angew. Botanik, Frankfurt, Sept., 1916. Chem.-Zeit., 1916, 40, 1073.

The normal annual production of potatoes in Germany is estimated at about 50 million metric tons. Assuming the loss through disease, freezing, etc., to be only 0.25%, it would be equivalent to at least 100,000 metric cwt. of starch. Experiments on a large scale have proved the practicability of utilising decomposed potatoes to obtain a sound, tasteless product, containing about 50% of starch and 6% of nitrogen, which has given good results in feeding experiments. The tubers may be treated either by a wet process (separation of starch by disintegration in water) or preferably by a dry process. With slight modification of the process it is also possible to separate the starch in the usual way, but for the preparation of pastes, dextrin, alcohol, etc., this is unnecessary, since the diseased potatoes may be directly hydrolysed with acid.—C. A. M.

*Fruits and vegetables; Conversion of — into dried products.* V. Valvassori. Atti. R. Accad. Econ.-agr., Florence, 1916, 13, 56—64. Bull. Agr. Intell., 1916, 7, 1353—1355.

In the case of pears and apples, the best results were obtained by subjecting the peeled and halved fruits to the action of sulphur dioxide for 10—15 mins., followed by treatment with steam for 5—10 mins., and then drying them at 80°—90° C. for 8 hours. Stoned peaches and apricots were sulphured for 15 mins. and then dried at 80°—90° C. for 8 hours. Plums were steamed for 90 secs., or immersed in boiling 5% potassium carbonate solution and then rinsed in cold water; they were dried first at 50° C., then at 70° C., and

finally at 90° C. Good results were obtained by either treatment. Cherries were steamed for 5 mins. and then dried at 85° C. for 12 hours, whilst figs were peeled, cut in halves, then treated with sulphur dioxide for 30 mins. and dried at 50° C. for 2 or 3 days. Vegetables, including potatoes, carrots, cabbage, cauliflower, spinach, celery, peas, beans, onions, leeks, turnips, and turnip tops were scalded or steamed and then dried at 40° to 60° C.—W. P. S.

*Cherry by-products; Utilization of —.* F. Rabak. U.S. Dept. Agric., Bull. No. 350, 1916. Bull. Agr. Intell., 1916, 7, 1355—1357.

ABOUT 1400 tons of cherry-stones and 112,000 galls. of juice are obtained as by-products in the cherry-preserving industry of the United States; these products are wasted at present but it would be quite possible to turn them into valuable commercial products. By treating the crushed stones with a solvent 8.3% of a fatty oil (pit oil) can be obtained, or, if the kernels alone are taken, and submitted to pressure, about 30% of fatty oil is obtained; the resulting cake, when steeped in water and steam-distilled, yields about 1% of a volatile oil. The oils have the following characters:—*Stone oil.* Sp.gr. at 25° C., 0.9137;  $n_D^{25} = 1.4641$ ; solidifying pt., 12° C.; saponif. value, 179.7; iodine value, 93.7; Reichert-Meissl value, 3.7; insoluble fatty acids, 92.5%; acetyl value, 3.45; unsaponifiable matter, 1.12%. *Kernel oil.* Sp.gr. at 25° C., 0.9092;  $n_D^{25} = 1.4635$ ; solidif. pt., 13° C.; saponif. value, 180.8; iodine value, 92.8; Reichert-Meissl value, 4.7; insoluble fatty acids, 92.8%; acetyl value, 12.67; unsaponifiable matter, 0.44%. *Volatile oil.* Sp. gr. at 4° C., 1.012; hydrocyanic acid, 7.94%; benzoic acid, 67.95%. The fatty oil does not differ essentially from sweet almond oil, whilst the volatile oil is, for all practical purposes, similar to that of bitter almonds. The pressed cake, after removal of the oils, is useful as a cattle food. It contains: Moisture, 1.05; proteins, 30.87; ash, 3.94; cellulose, 8.90; ether extract, 13.10%. The juice from the stones is a bright red liquid; it may be concentrated, after neutralisation with lime and filtration, to a syrup, or fermented to produce alcohol. With the addition of pectin and sugar, the concentrated juice forms an excellent jelly.—W. P. S.

[*Japanese*] seaweeds. Miyama. See VII.

*Analysis of honey and other substances containing lavulose.* Atkins. See XVII.

*Relationship existing between oxydase activity of plant juices and their hydrogen ion concentration, with a note on the cause of oxydase activity in plant tissue.* Bunzell. See XVIII.

#### PATENTS.

*Food products; Process of making [fatty] —.* T. H. Walker, Austin, Tex. U.S. Pat. 1,206,954. Dec. 5, 1916. Date of appl., Dec. 16, 1914.

A PRODUCT containing about 22% of stearin is made from an edible fatty material by hydrogenation, and then incorporated with a different edible fatty material to impart a flavour and consistency resembling those of a natural edible fat. A product resembling lard may thus be prepared from hydrogenated cottonseed oil and oily lard.—J. H. L.

*Alimentary pastes and other products; Drying chamber for —.* G. Falchi. Fr. Pat. 480,975, Feb. 15, 1916. \*

THE paste, e.g., macaroni, etc., is placed on trays.

or hung on supports arranged on a wheeled carriage and the latter is run into a closed chamber; air is pumped into the chamber and the current is distributed by fans; adjustable ventilators are provided for the escape of the air so that the rate of drying may be regulated.—W. P. S.

*Casein; Process for treating* —. A. Bartels, Harburg, Germany. U.S. Pat. 1,209,539, Dec. 19, 1916. Date of appl., Dec. 28, 1915. Renewed Aug. 16, 1916.

SEE Eng. Pat. 13,203 of 1915; this J., 1916, 1125.

*Artificial milk; Process for the manufacture of —, and treatment of its residues* —. W. J. Melhuish, Upper Parkstone. U.S. Pat. 1,210,667, Jan. 2, 1917. Date of appl., Oct. 22, 1915.

SEE Eng. Pat. 13,903 of 1915; this J., 1916, 1031.

*Separation of casein and albumin from creamery sewage*. Eng. Pat. 18,023. See XIXa.

### XIXa.—WATER PURIFICATION; SANITATION.

*Oxygen [in water]; Determination of — by Winkler's method*. G. Bruhns. Chem.-Zeit., 1916, 40, 985—987, 1011—1013. (See this J., 1916, 138.)

DETAILS of further small improvements in Winkler's process for the determination of dissolved oxygen are given.  $N/100$  Thiosulphate was found to be very satisfactory for titration and to keep well, particularly if made slightly alkaline to phenolphthalein. In the process, after precipitation has been effected, if the liquid completely clarifies upon standing, the greater part may be siphoned off and only the residue treated with potassium iodide and acid; the result is slightly more accurate owing to the small volume of liquid titrated. Long standing, however, is not desirable as the result may be affected by impurities in the water. When potassium bicarbonate is used, some gas bubbles are observed in the liquid after the crystals have dissolved; these bubbles are due to contraction of the liquid and are without influence on the result. The blue colour which reappears a short time after titration is due to delayed decomposition of the iodine-potassium iodide complex; the error thus caused does not, however, amount to more than 0.006—0.008 "per 1000 normal" (*loc. cit.*). A little additional blue colour may be produced subsequently owing to the catalytic action of manganese salts, the action of light, and the presence of nitrites.—J. H. J.

*Boiler-feed water; Determination of sodium and calcium bicarbonates in —*. M. Monhaupt. Chem.-Zeit., 1916, 40, 1041—1043.

THE case of a boiler water is considered which contains only bicarbonate alkalinity and is therefore neutral to phenolphthalein. The determination consists of two titrations with  $N/10$  acid in presence of methyl orange, firstly directly on the water, secondly after addition of  $N/10$  caustic soda equal to the acid used and removal of the precipitate by filtration. The acid used in the first titration is subtracted from that used in the second, and the result multiplied by 8.4, gives the amount of sodium bicarbonate in mgrms. per 100 c.c., if 100 c.c. of the water was taken. The acid used in the second titration is subtracted from twice that used in the first, and the result multiplied by 2.8 gives the amount of lime in the form of bicar-

bonate. Should there be a trace of phenolphthalein alkalinity in the water, this is titrated with the acid, and twice the result is subtracted from the second titration before calculating the sodium bicarbonate content. Calcium sulphate can only be present in the absence of sodium bicarbonate, and in this case twice the phenolphthalein alkalinity must be subtracted from the first titration before calculating the lime content. Salts of magnesium cause no irregularities in the method.—J. H. J.

*Tobacco smoke; Removal of nicotine from —*. J. Töth and K. Dangelmajer. Chem.-Zeit., 1916, 40, 1013.

It was found that many tobaccos yield a smoke containing large quantities of free nicotine, which can be extracted by passing the smoke through a cotton-wool plug treated with tannin. In some cases as much as 12.3% of the total nicotine can be recovered in this way. It is suggested that the use of such plugs by smokers would be of advantage from a health point of view.—J. H. J.

*Flavine and Brilliant Green: new and powerful antiseptics*. Browning and others. See XX.

*Use of diphenylamine and diphenylbenzidine for colorimetric determinations*. Smith. See XXIII.

### PATENTS.

*Sewage; Method of and apparatus for the treatment and purification of —*. E. B. Martin, Eccles, Lancs. Eng. Pat. 17,403, Dec. 14, 1915.

THE apparatus consists of a cylindrical tank, divided into segments, each segment forming a treatment unit. The apparatus may be worked on the continuous flow or on the quiescent system, or on a combination of both. The sewage enters any one segment where it meets with activated sludge, and the mixture is agitated by air blown in from the bottom and evenly distributed by baffle-plates. The treated sewage overflows into a radial trough in which any sludge settles out and is run back into the segment, while the liquid flows over a weir and is led into the next segment for further treatment. One or more of the last segments is used as a settling tank before the completely purified liquid is discharged. One segment is always being emptied and cleaned in the cycle of working. The activated sludge is run out from each segment into a central well. The volume of air supplied to any segment can be varied at will by adjustable valves.—J. H. J.

*Creamery sewage; Separation of casein and albumin from —*. G. Ebrill and T. Kiersey, Dublin. Eng. Pat. 18,023, Dec. 24, 1915.

CREAMERY sewage, i.e., any waste water containing milk or milk constituents, is treated with sulphuric acid (about 4.5 lb. of acid per 1000 lb. of skimmed milk contained in the sewage) and then led into a tank where it is aerated. The precipitated casein floats on the surface of the liquid and is discharged over the side of the tank into a collecting channel, an inclined baffle-plate being provided to guide the frothy precipitate over the side of the tank. The acidified liquid may be passed through a series of such aerating tanks. To remove albumin, the liquid, from which the casein has been separated, is treated with lime and conducted through a second similar series of aerating tanks. The aerating means are situated at the bottom of the tanks and consist of porous carbon tubes through which the air is forced so that it issues in a finely divided state.—W. P. S.

*Sewage; Process of disposing of* — A. Potter, New York. U.S. Pat. 1,207,821, Dec. 5, 1916. Date of appl., Oct. 18, 1913.

THE sewage flows at a very slow rate through a tank of such shape as to facilitate the deposition of the sludge, which is allowed to digest, and is then raised, a part allowed to flow away with the liquid from the tank, and a part allowed to return to the sludge space.—J. H. J.

*Water-softener; Process for producing a* — G. H. Widner, Assignor to Des Moines Refining Co., Des Moines, Iowa. U.S. Pat. 1,207,826, Dec. 12, 1916. Date of appl., Aug. 10, 1916.

A MINERAL containing hydrated aluminium silicate is dried to remove free moisture, ground to a suitable size, mixed with salt, and baked without fusing until hard enough to withstand the powdering action of water. It is then stored in water.—J. H. J.

*Water; Process and apparatus for softening* — L. Linden. Fr. Pat. 480,408, Dec. 13, 1915.

THE water is conducted into the upper part of a vertical cylinder containing perforated baffle-plates, certain of which are of copper and are connected with a source of electric current; carbon dioxide is also admitted to this part of the apparatus. The water then passes through filters (perforated metal plates supporting a layer of fabric and a layer of coke) placed across the cylinder, and is next distributed over the surface of horizontal plates in the lower half of the cylinder supporting layers of spongy aluminium. Some of these plates are connected with a source of electric current, and if necessary, carbon dioxide may be introduced. The water finally passes through a spongy iron filter and is collected at the bottom of the cylinder.—W. P. S.

*Disinfectant, antiseptic, deodorant, and detergent materials.* C. Billing, New York. Eng. Pat. 16,410, Nov. 20, 1915.

THE material consists essentially of a mixture of 9 parts of an alkali monoborate with 1 part of an alkali diborate, with or without sodium chloride. A fluid form is prepared by dissolving 136.24 parts by weight of caustic potash (90%) and 145.8 parts of boric acid in 150 parts of water, and when the solution is cold, adding water until the specific gravity is 1.42 at 60° F. (15.5° C.). Chlorides or hypochlorites may be added. A solid form is prepared by dissolving 89 parts by weight of caustic soda (70%) in 90 parts of water, adding 438 parts of borax, and heating with stirring until fluid; then allowing to cool, and continuing the stirring, until granular. About 116 parts of sodium chloride is then added, so that the product shall contain 40% of anhydrous borate.—J. H. J.

*Tins; Treatment of waste* — in a dust-destroyer. E. J. Lovegrove, London. Eng. Pat. 102,693, June 15, 1916. (Appl. No. 8449 of 1910.)

A CHAMBER for the treatment of the tins is placed beside a dust-destroyer, so that the whole or a portion of the flue gases can be passed through it. The chamber consists of a furnace with hopper at top, an entrance for flue gases below the hopper, and an exit at the bottom of the chamber. On each side wall below the grate, two plates are fixed, sloping towards a trough in the centre. The tins are fed into the chamber through the hopper, which is then closed, and the dampers controlling the gas pipes are opened to allow the flue gas to pass down through the mass of tins, between the sloping plates below the grate, and out. Dirt attached

to the tins is burned off, the solder is melted, drops on to the sloping plates, and runs into the trough. When the solder is all collected, the inlet pipes for flue gases are closed, the trough of solder withdrawn, and the tins removed through a side door.—J. H. J.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Flavine and Brilliant Green: new and powerful antiseptics.* C. H. Browning, R. Gulbransen, E. L. Kennaway, and L. H. D. Thornton. Report to the Medical Research Committee. British Medical J., Jan. 20, 1917. Pharm. J., 1917, 98, 73—74.

IN the case of all antiseptics in common use, a concentration which is sufficient to cause death of the organisms is also detrimental to phagocytosis. Thus, phenol kills organisms and inhibits phagocytosis at a concentration of 1:250 to 1:500; and mercury perchloride exerts both effects at 1:7,000 to 1:10,000. On the other hand, Brilliant Green kills cocci at 1:30,000 and only inhibits phagocytosis at 1:2,000, whilst Flavine kills both cocci and *B. coli* at a concentration of 1:100,000, whereas to affect phagocytosis a concentration greater than 1:500 is required. Of all the compounds examined, Flavine stands out as possessing the highest sum of desirable properties for therapeutic purposes, and tested clinically, the results as regards both Flavine and Brilliant Green have been eminently satisfactory, and the indications are that they will largely supersede the toxic antiseptics at present employed. Flavine (diamino-methyl-acridinium-chloride), which is likely to be the more generally useful of the two, was originally prepared by Benda (see this J., 1912, 878), and was found to have a very marked effect on trypanosome infections. Attention was first drawn by Browning and Gilmore to the powerful action of this substance on bacteria. On account of its trypanocidal action the compound was called Trypoflavine, but as its range of use promises to be much wider it is more convenient to denote it simply as Flavine. It is a fairly stable substance, and solutions may be boiled or heated up to 120° C. in the autoclave. Hitherto the preparation of Flavine has been worked out by Drs. Barger and Ewins in the chemical laboratory of the department of biochemistry and pharmacology of the Medical Research Committee, but arrangements have now been made for the commercial production of the compound on a larger scale. The products will be tested biologically at the Bland-Sutton Institute of the Middlesex Hospital, and will be available for trial on application to Dr. Browning on behalf of the Medical Research Committee.

*Alloxan; Preparation of* — H. Biltz and M. Heyn. Annalen, 1916, 413, 69—67.

WHILST chlorine has no action on uric acid suspended in boiling acetic acid, oxidation to alloxan occurs in the presence of three or more mols. of water, and by leading a rapid stream of chlorine through a mixture of 300 c.c. of glacial acetic acid and 50 c.c. of water in which 100 grms. of finely divided uric acid was suspended, a yield of 60—65 grms. of alloxan monohydrate was obtained after about 15 mins. chlorination, the completion of the reaction being indicated by the passing into solution of the uric acid and the deposition of coarse heavy crystals of the alloxan monohydrate. By recrystallisation from water, and, if crude uric acid has been employed, decolorisation with

animal charcoal, colourless crystals of alloxan tetrahydrate are obtainable. The preparation of alloxan by this method is sufficiently rapid to be adapted to a lecture experiment.

As an addendum to the paper the microscopic identification of the methylated uric acids by the crystal form obtained by precipitation from solution in concentrated sulphuric acid by dilution with water is recommended.—G. F. M.

*Urethane; Influence of acid radicles containing different alkyl groups on the narcotic action of —.* J. Odaira. Mem. Coll. Sci., Kyoto Imp. Univ., 1916, 1, 319—340.

Numerous acyl derivatives of urethane were synthesised by the action of the corresponding acid chlorides on this substance, and their physiological action was compared by experiments on rabbits and frogs. The narcotic action of urethane was strengthened by the acylation of the amino-group, and more particularly so when the acid residue contained a secondary alkyl group. Thus diethylacetylurethane, or detonal, and ethyl-propylacetylurethane, or epronol, are both more effective than the parent substance, and epronol is almost equal to veronal (diethylbarbituric acid) and hedonal (methylpropylcarbinol) urethane in the duration of narcotic action, and in addition much more rapid in action. It seems probable that a further increase in the number of carbon atoms up to propylbutyl- or dibutyl-acetylurethane still further increases the strength of the narcotic action. The minimum effective dose of epronol, which was studied more closely than the other substances, was 0.3—0.5 gm. per kilo. of body weight for a rabbit, whilst 2.5—3.0 grms. caused death in 20—50 hours. In doses of 1.5 grms. per kilo. the blood pressure remained normal, and the vagus and respiratory centres were not affected, but the action on the heart becomes more and more effective with the higher homologues as the narcotic action increases. N-Iso-amylurethane, which differs from detonal only by the absence of the carbonyl group, although a somewhat stronger narcotic, is far more poisonous than detonal.—G. F. M.

*Cyanamide from calcium cyanamide; Preparation of —.* E. A. Werner. Chem. Soc. Trans., 1916, 109, 1325—1327.

THE stability of cyanamide in the presence of acetic acid has been utilised to prepare it from calcium cyanamide, 100 grms. of the latter being added, in portions of 15 grms. each, to 125 grms. of pure acetic acid and 120 grms. of water in a cooled mortar with constant stirring. The pasty mass is well kneaded, and dried in the air for 24 hours. The easily-powdered solid is then extracted six or seven times with ether in a Soxhlet, the extract concentrated by distillation at a gentle heat, and finally evaporated to dryness in a desiccator over sodium hydroxide. The yield varied from 94 to 98% of the theoretical value. It is of great importance that the mass, before extraction with ether, be faintly acid throughout.—B. N.

*Saurol, a substitute for ichthyol.* L. Méran. Vet. Rec., 1916, 287. Pharm. J., 1917, 98, 43.

SAUROL is an oil obtained by distilling a bituminous shale found in a mine not far from Lake Lugano, Switzerland. The purified oil contains 6—7% of sulphur in organic combination and closely resembles ichthyol in its therapeutic properties.

*Sodium citrate; Action of — and its decomposition in the body.* W. Salant and L. E. Wise. J. Biol. Chem., 1916, 23, 27—58.

SODIUM citrate is rapidly oxidised and eliminated

when injected intravenously into cats and dogs; large doses when given by the mouth render the urine alkaline, but the urine contains only traces of the citrate. The fatal dose of sodium citrate, when injected intravenously, varies from 0.4 to 1.6 gm. per kilo. of animal, and 70 mgrms. may produce slight symptoms. Only large amounts (more than 3 grms. per kilo.) are toxic when ingested. The toxicity of sodium citrate depends on the rate of its oxidation in the body; it is more toxic for animals in which larger quantities are eliminated unchanged; the rate of oxidation is greater in rabbits than in cats. The presence of 1% of sodium citrate inhibits the coagulation of blood for 4 days at 10° C. The acute effect of sodium tartrate is less than that of sodium citrate.—W. P. S.

*Utilisation of cherry by-products.* Rabak. See XIXa.

*Estimation of arsenic in organic compounds.* Ewins. See XXIII.

#### PATENTS.

*Acetic anhydride; Manufacture of —.* H. Dreyfus. Basle, Switzerland. Eng. Pat. 17,920, Dec. 22, 1915. Under Int. Conv., Dec. 23, 1914.

ACETIC anhydride is manufactured by acting on an acetate with sulphuric anhydride (which may or may not be mixed with chlorosulphonic acid), or with a compound of sulphuric anhydride and an inorganic salt, e.g., sodium chloride or sulphate. A diluent such as acetic anhydride is used, and the reaction mixture must be cooled initially to 0°—5° C. if sulphuric anhydride is employed directly, or to atmospheric temperature by water-cooling if a compound of sulphuric anhydride is employed. *Example.* 800 kilos. of sulphuric anhydride is added fairly quickly, with constant stirring, to 1600 kilos. of powdered anhydrous sodium sulphate, and when combination is complete the mass is cooled and added to a mixture of 1640 kilos. of powdered anhydrous sodium acetate and 1600 kilos. of acetic anhydride, the mixture being stirred and water-cooled. The temperature may afterwards be allowed to rise to 60°—70° C. to complete the reaction, and the acetic anhydride is distilled off *in vacuo*. The product is of a high degree of purity, and free from chlorine.—F. Sr.

*Liniment.* C. A. and G. Cofman-Nicoresiti, London. Eng. Pat. 18,060, Dec. 28, 1915.

SOLID preparations of volatile oils such as amber oil, eucalyptus oil, camphor oil, terebene, etc., containing from 80% to 95% of the oil, are obtained by incorporating with the oil a salt of one of the higher fatty acids, such as stearic and palmitic acids. The salt as such may be emulsified with the oil, or separate portions of the oil may be mixed with the acid and base respectively and then mixed together. The addition of about 1 part in 500 of paraffin wax assists the solidification.—B. V. S.

*Pharmaceutical products containing arsenic, and process of making same.* J. Calisen, Elberfeld, and W. Grüttemann Vohwinkel, Germany. Assignors to Synthetic Patents Co., Inc., New York. U.S. Pat. 1,201,692, Oct. 17, 1916. Date of appl., July 29, 1916.

COMPOUNDS containing the following atomic groups are claimed:— $\text{RO}$ ;  $\text{CR}'\text{As}$ ,  $\text{ROX}$ ;  $\text{CR}'\text{As}$ ,  $\text{ROX}$ ;  $\text{CR}'\text{AsO}$ ;  $\text{RCX}$ ;  $\text{CR}'\text{AsO}(\text{OH})$ ,  $\text{ROCl}$ ;  $\text{CR}'\text{AsO}(\text{OH})$  (in which R, R' is hydrogen or a hydrocarbon

radical, and X is a halogen), and in particular the chloroarsinic acids produced by causing arsenic trichloride to combine with acetylene hydrocarbons, treating the product with water to form the arsenoxide, and oxidising the latter. *Example:* 240 parts of heptene (*n*-pentylacetylene) and 900 parts of arsenic trichloride are heated together for 16 hours under a reflux condenser. The excess of arsenic trichloride is removed by distillation *in vacuo*, and the oily residue is dissolved in 3000 parts of wet ether. Aniline is added as long as a precipitate forms, and the aniline hydrochloride is filtered off. The filtrate is washed with dilute hydrochloric acid, then with water until neutral to Congo red, dried over sodium sulphate and freed from ether *in vacuo*. Heptenechloroarsenoxide remains as a thick dark liquid. 41 parts of heptenechloroarsenoxide is dissolved in 400 parts of acetone and treated with 3% hydrogen peroxide until oxidation is complete. The solution is shaken with 500 parts of ether, and the ethereal layer washed with dilute caustic soda and water. The alkaline wash water is acidulated with hydrochloric acid, and evaporated *in vacuo* at 60°–70° C. until the heptenechloroarsinic acid crystallises. It is separated, washed with a little ether, heated with animal charcoal in 50 parts of water, and recrystallised. It forms white shining leaflets, easily soluble in water to a neutral solution. It is valuable in the treatment of anaemia, chlorosis, leucæmia, chorea, and skin diseases.—F. Sp.

*Auro-mercaptobenzenes, and process of making same.* A. Feldt, Frankfurt, and P. Fritzsche, Assignors to Farb. vorm. Meister, Lucius, und Brünig, Höchst, Germany. U.S. Pat. 1,207,284, Dec. 5, 1916. Date of appl., Oct. 17, 1916.

AURO-MERCAPTOBENZENES, of the general formula, X.S.Au, where X is any benzene nucleus, are obtained by the action of double gold chlorides on mercaptobenzenes. They are yellowish solids. The sodium salt of 4-amino-2-auromercaptobenzene-1-carboxylic acid is a yellowish powder, readily soluble in water, insoluble in organic solvents.—B. V. S.

*Halogenation of hydrocarbons and other processes of chlorination, and reactions between gases or vapours in general.* B. S. Lacy. Fr. Pat. 480,064, Oct. 22, 1915.

SEE Eng. Pat. 14,709 of 1913; this J., 1916, 653. The method is applied to other processes of halogenation and to reactions between gases or vapours in general.

*Tobacco-plants; Process for treating the green juice of —.* T. Oelenheinz, Karlsruhe, Germany. U.S. Pat. 1,209,327, Dec. 19, 1916. Date of appl., July 24, 1912.

SEE Eng. Pat. 17,037 of 1912; this J., 1913, 882.

*Olefinic terpene derivatives; Hydrogenising —.* C. Paal, Leipzig, Germany. U.S. Pat. 1,210,681, Jan. 2, 1917. Date of appl., July 18, 1914.

SEE Eng. Pat. 16,180 of 1914; this J., 1915, 982.

*Alcohols; Manufacture of [aliphatic] —.* C. Dill. Fr. Pat. 477,812, Oct. 6, 1914. Under Int. Conv., Oct. 4, 1913.

SEE Eng. Pat. 20,550 of 1914; this J., 1915, 575.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Colour sensitising of photographic plates; Preparation of dyes for —.* W. Harrison and S. E. Bottomley. Preliminary Report of the Investigation carried out at the University of Leeds and the City of Leeds Technical School (Printing Crafts Department). Nov. 1916.

METHODS are given for the preparation of ethyl iodide, quinaldine (Doebner and Miller, Ber., 1883, 16, 2465), toluquinoline (Skraup, Monatsh. Chem., 1881, 2, 158), and toluquinaldine, and from these of Formocyanine (Pinacyanol) (Ger. Pat. 172,118; see Eng. Pat. 16,227 of 1905, this J., 1906, 368), and Toluocyanine (Orthochrome T) (Ger. Pat. 167,159; see Eng. Pat. 9598 of 1903; this J., 1904, 384). Small quantities of the dyes in an impure condition have been prepared; spectra of plates prepared with Formocyanine prepared as described, and with Pinacyanol of German origin are given for comparison.—B. V. S.

*Colour sensitising; Production of dyes for —.* W. J. Pope. Brit. J. Phot., Jan. 12, 1917.

IMPROVED methods have been devised at the University Chemical Laboratory, Cambridge, for preparing the intermediate products and all the sensitisers in ordinary use, including Pinaverdol, Pinacyanol, and Orthochrome, and several plate-makers have been supplied with these dyes for some months. Details of the methods in use are not given.—B. V. S.

## PATENTS.

*X-Ray photography.* T. T. Baker, South Croydon. Eng. Pat. 17092, Dec. 4, 1915.

THE time of exposure in taking X-ray photographs is reduced by the use of two intensifying screens, on either side of the sensitive plate or film, the one nearer the X-ray tube being of crystalline calcium tungstate, more transparent to X-rays, and the other one being of the semi-crystalline salt, much less transparent to X-rays.—B. V. S.

*Developing and fixing photographic exposures; Composition of matter for —.* F. W. Hochstetter, Assignor to H. P. Patents and Processes Co., Inc., New York. U.S. Pat. 1,207,042, Dec. 5, 1916. Date of appl., Oct. 7, 1913.

A COMBINED developing and fixing solution contains a reducer, sodium sulphite and thiosulphate, an alkali carbonate, glycerin, and citric acid.—B. V. S.

*Photographic process. [Colour photography.]* W. F. Fox, Assignor to Kinemacolor Co. of America. New York. U.S. Pat. 1,207,527, Dec. 5, 1916. Date of appl., June 23, 1914. (See also Eng. Pats. 552 and 8728 of 1914; this J., 1914, 1227.)

TWO negatives are taken through screens of complementary colours (e.g., red and green). A print is made through one of the negatives and toned to a colour complementary to that of the taking screen. A second print, through the other negative, is then superposed in register on the first print and stained by mordanting and dyeing to the colour complementary to its taking screen.—B. V. S.

*Photographs and photogravures; Process for producing*—J. H. Christensen. Fr. Pat. 481,149, Mar. 10, 1916. Under Int. Conv., Mar. 10, 1915.

AFTER development plates are treated with a fixing solution which acts on the unreduced silver salt to form a compound which fills up the pores of the film. The most suitable substances to use are alkali polysulphides and thioannates, Schlippe's salt, and similar compounds which easily deposit sulphur in the fixing process, but similar effects can be obtained with iodides, thiocyanates, and some other substances. The best effect is obtained by a mixture of a sulphur compound and a solvent of silver halides such as thio-cyanate. Collodio-bromide films to be treated in this way are preferably previously made very porous by the addition of a substance such as glycerin, benzoic acid, etc., which washes out during development, while gelatin films should be previously hardened with chrome alum, alum, or formaldehyde. The varying porosity of the films on plates so prepared may be utilised in several printing processes. For example, colour printing plates for a three-colour process may be prepared by printing through the colour negatives on to a composite plate consisting of a lower dyed collodion film on a suitable base and an upper collodio-bromide film. After development and fixation according to this process, the transfer of dye from the lower film to a printing paper varies with the porosity of the upper film, and a coloured print is obtained.—B. V. S.

*Colour photography.* P. D. Brewster, East Orange, N.J. U.S. Pat. 1,208,739, Dec. 19, 1916. Date of appl., Jan. 29, 1914. Renewed May 11, 1916.

SEE Eng. Pat. 1073 of 1915; this J., 1916, 329.

## XXII.—EXPLOSIVES; MATCHES.

*Trinitrotoluol; U.S. Navy Department Specifications for*—

THE following U.S. Navy Department Specifications for trinitrotoluol (51T2) have been issued:—

*Grade A—Refined.* Trinitrotoluol must be a high-grade material, made from a suitable quality of raw materials. It must be thoroughly purified by recrystallisation from an approved solvent, so that the finished product shall have the following characteristics:—The material must be in the form of a slightly yellow, fine, and uniform crystalline powder. At least 99% must pass through a sieve 12 mesh per linear inch. No odour of any by-product or crystallising agent may be present. It shall have a melting point of at least 79° C. It shall be free from acidity and shall not show more than traces of metallic salts or residual solvents. Average moisture of a lot shall not be greater than 0.1%. Average ash of a lot shall not be greater than 0.1%. It shall not contain more than 0.15% of material insoluble in alcohol. It shall contain no free toluol or products giving a nitric-acid reaction with a sulphuric acid solution of diphenylamine. It shall contain not less than 18.20% nitrogen as determined by the Dumas combustion method. It shall give a heat test of at least thirty minutes at 65.5° C. with potassium iodide-starch paper.

*Grade B—Crude.* Trinitrotoluol must be a high grade material, made from a suitable quality of raw materials. It shall be thoroughly stabilised, and washed free from stabilising materials and by-products of stabilising process, and thoroughly dried. It shall be a yellowish, uniform crystalline powder of such fineness that at least 90% will pass

a sieve 10 meshes per linear inch. It shall have a melting point of at least 75.5° C. It shall be free from acidity. It shall show not more than traces of metallic salts. Conditions as to ash, moisture, percentage soluble in alcohol, and content of free toluol or products giving a reaction with diphenylamine are the same as for the refined product. It shall contain not less than 18% nitrogen as determined by the Dumas combustion method.

The manufacture of the material shall be open to inspection in all its details by the officers and employees of the Bureau of Ordnance assigned to duty for that purpose, and must be satisfactory at all its stages to the inspector at the works or to such one of his assistants as may be designated.

For purposes of inspection 4000 lb. of trinitrotoluol will constitute a "lot," and from each lot the inspector will select a sample of one pound and ship it at the expense of the Government to the Naval Proving Ground, Indian Head, Md., for chemical test.

*Additive compounds of trinitrobenzene.* Sudborough. See III.

*Additive compounds of a-trinitrobenzene with amino-derivatives of hydrocarbons.* Cadre and Sudborough. See III.

### PATENTS.

*P perchlorate explosives.* Stockholms Superfosfat Fabriks Aktiebolag, Stockholm. Eng. Pat. 17,584, Dec. 10, 1915. Under Int. Conv., Dec. 23, 1914.

EXPLOSIVES containing perchlorates are subjected to a pressure of not less than 300 kilos. per sq. cm., which diminishes their sensitiveness to shock and increases their specific gravity, thereby making them suitable for charging shells.—F. Se.

*Nitrocellulose; Process of producing*—A. C. Vournasos, Athens. Eng. Pat. 9547 of 1915; date of appl., July 22, 1914.

NITROCELLULOSE is produced from cellulose derived from *Hibiscus cannabinus* (see previous abstract) by immersing 30 parts by weight of the fibres in a mixture of 250 parts of nitric acid (sp.gr. 1.38) and 500 parts of sulphuric acid (sp.gr. 1.82) for 24 hours at 15° C. Either the crude or the bleached fibres may be used, the latter yielding a product stated to be superior in stability to the nitrocellulose from pure cotton.—F. Se.

*Matches [; Damp-proof*—W. Mills, Birmingham, and W. Morgan, Bristol. Eng. Pat. 18,028, Dec. 28, 1915.

MATCHES tipped with brimstone or other inflammable material are rendered damp-proof by coating them with a solution of celluloid or nitrated cellulose. In addition to this treatment, the solution may also be applied to the wood prior to the application of the brimstone, etc.—C. A. M.

*Match-ignition material. Process of drying the composition heads of matches.* W. A. Fairburn, Short Hills, N.J., Assignor to The Diamond Match Co., Chicago, Ill. U.S. Pat. (A) 1,206,827 and (B) 1,206,828, Dec. 5, 1916. Dates of appl., Apr. 6, and July 28, 1915.

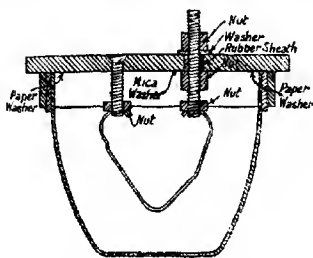
(A) Rosin, dextrin, plaster of Paris, antimony sulphide, and amorphous phosphorus are incorporated to form a match-ignition material. (B) An oxidising composition is applied to the match splints, which are then passed through a series of drying zones, where they meet rapid currents of fairly dry air, first at temperatures

increasing successively to about 120° F. (49° C.) and then decreasing successively to about 80° F. (27° C.), after which the heads are coated with the sensitive ignition composition.—C. A. M.

### XXIII.—ANALYSIS.

*Combustion methods for use in the laboratory.*  
[Determination of sulphur.] J. Hewett.  
London and Southern Distr. Jun. Gas Assoc.,  
Jan. 5, 1917. Gas J., 1917, 137, 72—73.

SULPHUR may be estimated in volatile liquids, e.g., crude benzols, by drawing air through the liquid and after further diluting the air-vapour



mixture, passing it through a silica tube heated to redness, when complete combustion occurs. The gases then pass through two wash-bottles containing sodium hydroxide. After oxidation of the product with bromine or hydrogen peroxide, sulphur is estimated as sulphate in the usual way. The author indicates the wide applicability of the estimation of sulphur by combustion with sodium peroxide. It is carried out in a nickel crucible (see fig.) fitted with a screwed top and electrical ignition device. A ring of filter paper makes a tight joint. The crucible may be immersed in water when the charge is fired. The substance under test must be thoroughly incorporated with the sodium peroxide in suitable proportions. The reaction should be complete in a few seconds. The contents of the crucible when cold, are extracted with distilled water and after acidification and filtration, sulphur is estimated as sulphate in the usual way. This method may be used for any substance which can be brought to react vigorously with sodium peroxide either alone or with the addition of sulphur-free combustible matter, e.g., wood dust or alcohol. It has been used with tar and tar products, oils, fats, greases, coal, coke, new and spent oxide of iron. With spent oxides, the reaction product is suitable for the estimation of iron and alumina as well as sulphur. The analysis of spent oxide in this way can be conducted much more speedily and conveniently than by the usual methods. The reaction is extremely violent. Not more than 1 gm. of combustible substance should be taken and then 20 grms. of sodium peroxide usually gives a suitable melt. The substance should be approximately free from moisture, as water may cause dangerous premature ignitions.—H. J. II.

*Calorimetry by combustions with sodium peroxide.*  
W. G. Mixer. Amer. J. Sci., 1917, 43, 27—34.

A DETAILED account of the construction of a silver bomb-calorimeter (and of the necessary appurtenances) employed by the author in a

series of thermochemical researches. Full directions are given for its use in the determination of the heat of oxidation of elements which do not burn in oxygen and which form oxides insoluble in acids. The method of fusion with sodium peroxide is also adapted to the determination of the heat of combination of certain oxides with sodium oxide. Experimental figures for determination of the heats of formation of  $\text{Fe}_2\text{O}_3$  and of pyrites are appended. Results obtained by this method are compared with those yielded by other modes of procedure. Some substances when burnt with  $\text{Na}_2\text{O}_2$  do not evolve sufficient heat to fuse the mixture and some readily combustible body such as sulphur or carbon must be added.—F. C.

*Phosphoric acid in qualitative analysis; Removal of —.* L. Gattermann and H. Schindhelm.  
Ber., 1916, 49, 2416—2422.

WORKING with 1 gm. of material, the filtrate from the hydrogen sulphide group is boiled until free from hydrogen sulphide, concentrated to 100 c.c., filtered if necessary, and treated with 5 c.c. of 6% ammonium phosphate solution. It is then nearly neutralised with dilute ammonia (until only faintly acid to litmus), 3 c.c. of dilute hydrochloric acid (7—8%) is added, and the brisly boiling solution is treated with 3 c.c. of a freshly prepared solution containing 3 grms. of crystallised stannic chloride, dissolved in the cold. Stannic phosphate is thus precipitated, and if necessary more of the stannic chloride solution is added, in portions of 0.5—1 c.c., to the boiling solution until no further precipitate is obtained on testing a little of the filtered liquid with ammonium molybdate. The boiling solution is then filtered, using a Büchner funnel and double filter, the precipitate washed with boiling water, the warm filtrate, which should be quite clear, is treated with dilute hydrochloric acid and hydrogen sulphide, to remove excess of tin, and the metals present are detected in the usual way. When the phosphate precipitate has a greenish colour, it is tested with caustic soda and hydrogen peroxide for chromium which, like ferric (but not ferrous) iron, is liable to be carried down with the precipitate. If too little phosphoric acid be present, the mixture containing the stannic phosphate precipitate is difficult to filter, but satisfactory conditions are ensured by adding ammonium phosphate as directed.—F. SOX.

*Diphenylamine and diphenylbenzidine; Use of — for colorimetric determinations.* L. Smith.  
Z. anal. Chem., 1917, 56, 28—42.

THE intensity of the colour obtained with diphenylamine in the colorimetric estimation of nitric acid (compare Tillmanns, this J., 1911, 44, 918), is largely influenced by shaking the tube, excessive shaking causing a diminution. To obtain reliable results, therefore, shaking should be avoided, the necessary mixing being done by moving a suitable stirrer a few times up and down in the tube, and then allowing the liquid to remain at rest for the prescribed period of one hour. It is suggested that the observed phenomenon is due to the formation of a volatile substance during the oxidation of diphenylamine, the presence of hydrogen chloride being also a determining factor. The stability of the colour is at a maximum at ordinary temperatures. The sensitiveness of the reagent depends largely on its composition, and for the determination of the following amounts of nitric anhydride per litre the annexed formulae are recommended:—0.1—3.0 mgrms.; 0.04 gm. diphenylamine, 39 c.c. water, concentrated sulphuric acid to 100 c.c.; 0.5—10.0 mgrms.; 0.04 gm. diphenylamine, 26 c.c. water, and sul-



phuric acid to 100 c.c.: 1.0–25 mgrms.; 0.08 gm. diphenylamine, 20.5 c.c. water, and sulphuric acid to 100 c.c. Diphenylbenzidine behaves analogously to diphenylamine as a reagent for nitric acid; its sensitiveness is somewhat greater. —G. F. M.

**Arsenic in organic compounds; Estimation of —.** A. J. Ewins. Chem. Soc. Trans., 1916. 109. 1355–1358.

THE methods, previously described for the estimation of arsenic in organic compounds, are either very laborious and involve complicated processes, or are only applicable to certain compounds, such as salvarsan or neosalvarsan. The Norton-Koch method (this J., 1905, 1129), the principle of which is the same as the Kjeldahl method of estimating nitrogen, has been successfully applied by the author to the estimation of arsenic in arsanilic, acetylarsanilic, and diethylbenzarsinic acids, salvarsan, and a number of other organic substances. 0.1–0.2 gm. of the substance is heated with 10 mgrms. of potassium sulphate, 0.2–0.3 gm. of starch, and 20 c.c. of concentrated sulphuric acid in a Kjeldahl flask of about 300 c.c. capacity for about 4 hours, until the liquid becomes colourless or pale yellow. The contents are cooled, made just alkaline with 10–12 N caustic soda, cooled to 30°–40° C., and the liquid made just acid with sulphuric acid. A saturated solution of sodium bicarbonate is added until an excess of 5–10 c.c. of this reagent is present, then 2 c.c. of a 1% solution of starch, and the arsenious acid is titrated with N/20 iodine, until a permanent deep blue colour is obtained. —B. N.

**Determination of naphthalene [in coal gas] by picric acid.** Knublauch. See IIA.

**Assaying gold in copper matte.** Chase. See X.

**Analysis of antimonial lead.** McCabe. See X.

**Copying ink pencils and the examination of their pigments in writing.** Mitchell. See XIII.

**Varnish analysis and varnish control. II. Viscosity of varnishes.** Seaton and others. See XIII.

**Manufacture of linoleum and its valuation.** De Waele. See XIII.

**Valuation of water-soluble as compared with citrate-soluble phosphoric acid.** Frank. See XVI.

**Determination of minute amounts of sugar by  $\alpha$ -naphthol and estimation of entrainment losses.** Blake. See XVII.

**Simplified inversion process for determination of sucrose by double polarisation.** Walker. See XVII.

**Action of different decolorising carbons on pure and impure sugar solutions, and their influence in the analysis of sugar factory products.** Pellet. See XVII.

**Report on sugar. [Modification of the Clerget process of double polarisation. Use of invertase as hydrolyst in the double polarisation method.]** Browne. See XVII.

**Determination of raffinose in presence of sucrose by double inversion, using top and bottom fermentation yeasts.** Pellet. See XVII.

**Methods for the estimation of mixtures of four or more carbohydrates, involving oxidation with bromine.** Wilson and Atkins. See XVII.

**Analysis of honey and other substances containing levulose.** Atkins. See XVII.

**Determination of free and combined galactose.** Van der Haar. See XVII.

**Determination of ammonia [from nitrogen in brewing materials] by the boric acid method.** Adler. See XVIII.

**Scientific control of a rum distillery.** Seard. See XVIII.

**Influence of carbohydrates on the accuracy of the Van Slyke method in the hydrolysis of casein.** Hart and Sure. See XIX.

**Protein-copper compounds.** Osborne and Leavenworth. See XIX.

**Determination of oxygen [in water] by Winkler's method.** Bruhns. See XIX.

**Determination of sodium and calcium bicarbonates in boiler-feed water.** Monhaupt. See XIX.

#### PATENT.

**Gas; Apparatus for the analysis of —.** Aktiebolaget Ingeniörsfirma F. Egnell. Fr. Pat. 480,661, Jan. 12, 1916. Under Int. Conv. Feb. 6, 1915.

IN apparatus for determining the proportions of carbon dioxide and monoxide, oxygen, and hydrocarbons in a gaseous mixture, the gas passes in series through a measuring vessel, an absorbing solution, and finally either in series or parallel through a retort containing electrically heated finely divided metal or oxide which reduces or oxidises the gas, and a measuring vessel for the residue of gas. In a modification, additional absorption apparatus may be inserted before and/or after the retort. In another modification, the retort may be placed before the first measuring vessel and another absorption apparatus after that vessel. The residue of gas may be cooled and humidified. —W. F. F.

### Books Received.

**YEAR-BOOK OF PHARMACY AND TRANSACTIONS OF THE BRITISH PHARMACEUTICAL CONFERENCE.** JULY 12, 1916. J. and A. Churchill, 7, Great Marlborough Street, London. 540 pages, large 8vo. Price 10s.

THE first section of this useful year-book, covering 432 pages, contains abstracts of the various papers on chemistry, pharmacy, and materia medica, edited by J. O. Braithwaite, together with chapters on new remedies, by T. Stephenson. The remainder of the book, edited by R. R. Bennett, is occupied by the Transactions of the British Pharmaceutical Conference at the 53rd Annual Meeting, held in London on July 12th, 1916. Only one paper is reproduced, namely, that on "Some medicinal plants of Australia," by S. Plowman.

**PROHIBITIONS OF EXPORT IN FORCE IN BRITISH INDIA, THE SELF-GOVERNING DOMINIONS, EGYPT, AND CERTAIN OTHER BRITISH POSSESSIONS.** Supplements to "The Board of Trade Journal," Part I., Jan. 18, and Part II., Jan. 25, 1917. Price 3d. each.

**TRADING WITH THE ENEMY.** Consolidating statutory list of persons and firms in countries, other than enemy countries, with whom persons and firms in the United Kingdom are prohibited from trading. Complete to Dec. 22, 1916. No. 15A. H.M. Stationery Office, Imperial House, Kingsway, London, W.C. Price 3d.

